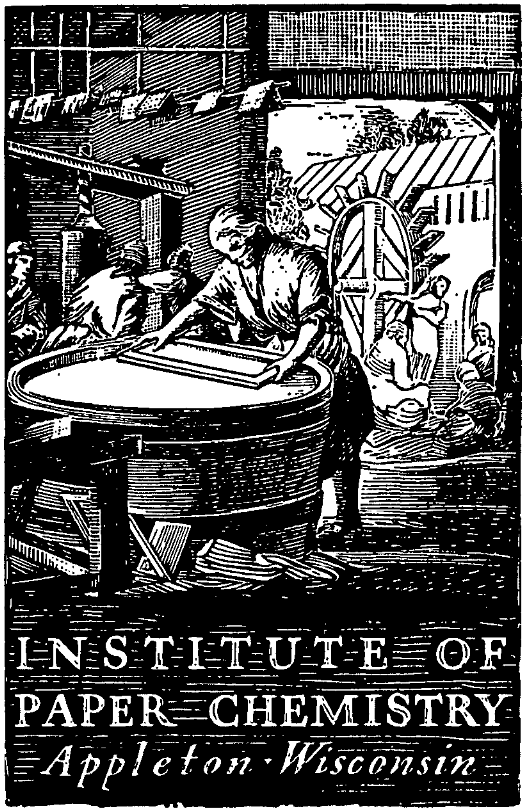
Carl Smith



FUNDAMENTAL STUDY OF ADHESION OF CORRUGATED BOARD

Project 2696-4

Report Four

A Progress Report
to
FOURDRINIER KRAFT BOARD INSTITUTE, INC.

July 21, 1971

GENERAL

THE INSTITUTE OF PAPER CHEMISTRY

Appleton, Wisconsin

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SUMMARY

The final segments of laboratory work concerned with optimization of the surface properties of corrugating medium for adhesion have been completed. Based on results obtained in exploratory studies, three samples of experimental medium were prepared for the adhesion study. Two of the samples were comprised of commercial semichemical mediums which were treated to provide a range in surface receptivity. One of the commercial mediums also provided relatively smooth and rough surfaces. The third sample consisted of handsheets prepared from redispersed commercial semichemical medium and dry lap softwood kraft pulp. The handsheets were treated to yield a range in receptivity and were wet pressed under conditions which produced relatively smooth and rough surfaces. Porosity varied from approximately 400 to 1800 ml./min. considering all three samples. The experimental mediums were attached to a 4-mil carrier sheet and subsequently corrugated at 150, 300, 450, and 600 f.p.m. utilizing a conventional two-step starch adhesive and a one-step starch adhesive.

The results show that optimum pin adhesion was attained at low-to-intermediate water drop values or, in other words, at high-to-moderate receptivities. Highly nonreceptive medium produced the poorest adhesion in all comparisons. There is some evidence to suggest that the optimum water drop value increases as the porosity of the medium increases or, in effect, a medium of low porosity requires a more receptive surface than one of high porosity. In the present work, optimum adhesion at low sheet porosity (480-560 ml./min.) was attained at water drop values of 34-53 seconds whereas optimum adhesion at intermediate

and high porosities (850 and 1800 ml./min.) was reached at water drop values of approximately 100 and 250 seconds, respectively.

In most cases where a direct comparison of relatively smooth and rough surfaces could be made on the same substrate, the smooth surface provided the better adhesive strength. An exception was obtained with the low-porosity handsheets when using the conventional two-step adhesive in which case the rougher surface showed some advantage at corrugating speeds up to 450 f.p.m. The aforementioned comparisons were made at smoothness levels typical of commercial medium. No attempt was made to test unusually smooth or glazed surfaces.

Comparison of the two starch adhesives showed an advantage for the one-step adhesive at corrugating speeds of 150, 300, and 450 f.p.m. on most surfaces. The advantage was not maintained at 600 f.p.m., presumably because the gel point of the adhesive was slightly higher than usual; however, it is expected that this problem could be remedied by adjustment of the alkali content.

The results obtained in this phase of the program were interpreted in terms of penetration theory and in terms of the results obtained earlier in the more fundamental study of adhesion (1, 2).

INTRODUCTION

The final phase of the program on Project 2696-4 is concerned with optimization of the surface chemical and surface physical properties of corrugating medium for adhesion. Initial efforts in this direction (3) involved preparation of experimental medium on the Institute's continuous web former. Medium was prepared which provided the desired range in receptivity and smoothness but the internal bonding strength proved weak and failure in the pin adhesion test occurred within the medium in most cases. While the information obtained from this study was limited in scope, the available data tended to confirm the importance of smoothness to adhesion in the corrugating operation as was indicated in earlier studies (1, 2). However, optimization with respect to receptivity was not achieved and it is with this subject that the current program is largely concerned. Since acceptable medium was not prepared on the web former, consideration was given to surface modification of commercial medium and to the use of handsheets. In pursuing this work, consideration was again given to those properties of the starch adhesive other than surface tension and viscosity which are believed to influence the extent of starch and water penetration, i.e., the size and uniformity of the swollen starch granules.

EXPERIMENTAL

EXPLORATORY STUDIES

Several series of exploratory tests were made to establish suitable conditions for modifying existing commercial medium and for preparing handsheets which would provide both the desired range in surface properties and adequate internal strength to withstand delamination in the pin adhesion test. The first series utilized a 70:30 blend of redispersed commercial waterleaf semichemical medium (Medium A) and dry lap softwood kraft pulp. The pulps were beaten separately in two steps and then combined to provide freeness levels of 480 and 330 cc. C.S.F. Handsheets equivalent to 26-lb. medium were prepared at pH 7.5-8.0 with and without sizing agent at each freeness level. Four sets of sheets were prepared under normal wet pressing conditions, i.e., five minutes at 50 lb. between blotters. One set was prepared under modified pressing conditions to provide relatively smooth and rough surfaces. This was accomplished by pressing between blotters for one minute to remove excess moisture and then between a polished metal plate and blotters for four minutes at 50 lb. pressure. The sizing agent used was Aquapel 360X (Hercules, Inc.) which was added in an amount equivalent to 0.1% based on fiber along with 0.1% of Kymene 557 (Hercules, Inc.) as a retention aid. The sheets were subsequently corrugated at 150 f.p.m. using a four-mil kraft carrier sheet and the conventional two-step starch adhesive. Results are summarized in Table I.

The pin adhesion test specimens from the first series showed a delamination or decapping type of failure including the set (No. 3) which had been pressed against the metal plate. This effect was similar to that previously found in medium produced on the continuous web former (3) and, hence, other means were sought to circumvent this problem.

TABLE I

PHYSICAL TEST DATA FOR HANDSHEETS FORMED FROM COMMERCIAL MEDIUM "A" AND FRESH KRAFT
(70:30 Blend Medium:Kraft)

Set No.	Description	Combined Freeness, C.S.F.	Caliper, mils	Bendtsen Porosity, ml./min.	Bendtsen Smoothness, ml./min.		Water Drop, sec.	Pin Adhesion, lb.		
					Smooth Side	Rough Side		Smooth Side	Rough Side	Type of Failure
1	Control	480	10.8	1850	2798	3000 +	22	87.0	77.0	Within medium (delamination)
2	Sized with 0.1% Aquapel 360X	480	10.4	1700	2906	3000 +	600 +	68.0	63.3	Within medium (delamination)
3	Control — pressed against plate	480	10.2	--	2243	2930	25	75.0	--	Within medium (delamination)
4	Control	330	10.4	450	2676	3000 +	67	95.7	83.3	Within medium (delamination)
5	Sized with 0.1% Aquapel 360X	330	10.2	450	2607	3000 +	600 +	71.3	68.0	Within medium (delamination)

Note: The corrugator was operated at 150 f.p.m.

The use of an existing commercial semichemical medium (Medium B) was examined in a second series of tests. The advantage of the commercial medium in this case was a definite two-sidedness with respect to smoothness. The medium was tested as is and after surface treating with 0.2% of Aquapel 364 from ether solution. Application was made by spraying a metered amount of the solvent solution on both sides of the medium. Because of the low boiling point of the solvent, the medium was not appreciably wetted in the surface applications. Pin adhesion results for medium corrugated at 150 f.p.m. are listed in Table II. The results obtained in this brief examination showed good correlation with surface smoothness and receptivity and the medium was strong enough to resist delamination.

A third series utilized both a commercial semichemical medium (Medium C) and handsheets prepared from the redispersed medium combined with dry lap softwood kraft pulp. For use in handsheets, the medium and kraft were beaten separately and then combined to provide a freeness of approximately 450 cc. C.S.F. Aquapel 360X was again used internally for increasing the water resistance of the handsheets and Triton X-100 (Rohm & Haas Co.) was applied to the surface of the sheets to decrease water resistance. The handsheets prepared at pH 7.5-8.0 were wet pressed at 75 lb. in contact with a polished metal plate as described in the first series. In utilizing the existing medium, receptivity was varied by spraying the surface with Triton X-100 from acetone solution and with Aquapel 364 from ether solution. The handsheets and commercial medium processed in this manner were corrugated at 150 f.p.m. using the 4-mil carrier sheet and the conventional starch adhesive. The results, which are recorded in Table III, indicate that the commercial medium and the handsheets prepared therefrom would meet the requirements of strength and surface properties.

TABLE II

PHYSICAL TEST DATA FOR COMMERCIAL MEDIUM "B"

Set No.	Treatment	Caliper, mils	Bendtsen Porosity, ml./min.	Bendtsen Smoothness, ml./min.	Water Drop, sec.	Pin Adhesion lb.	Pin Adhesion Type of Failure
6	None — rough side	9.9	1750	2650	13	72.7	Within medium and liner
7	None — smooth side	--	1770	2290	13	74.7	Within liner
8	Surface treated with 0.2% Aquapel 364, rough side	--	--	--	600 +	41.0	Within medium and at medium-adhesive interface
9	Surface treated with 0.2% Aquapel 364, smooth side	--	--	--	600 +	48.0	At medium-adhesive interface

Note: The corrugator was operated at 150 f.p.m.

TABLE III

PHYSICAL TEST DATA FOR COMMERCIAL MEDIUM "C" AND HANDSHEETS PREPARED THEREFROM

Set No.	Description	Additives or Surface Treatments, % based on fiber	Caliper, mils	Bendtsen Porosity, ml./min.	Bendtsen Smoothness, ml./min.		Water Drop, sec.	Pin Adhesion[a]	
					Smooth Side	Rough Side		lb.	Type of Failure
10	Medium "C" — Control	None	9.7	837	3000 +	3000 +	70	64.8	Within liner
11	Medium "C"	Triton X-100, 0.1	--	--	--	--	24	49.7	At liner-adhesive interface
12	Medium "C"	Aquapel 364, 0.2	--	--	--	--	600 +	54.7	At liner-adhesive interface
13	Handsheets from 70:30 blend of Medium "C" and fresh kraft	None	9.6	482	2448	3000 +	70	52.0	Within liner
14	Handsheets from 70:30 blend of Medium "C" and fresh kraft	Aquapel 360X, 0.1 Kymene 557, 0.1	9.8	507	2286	3000 +	600 +	42.7	At medium-adhesive interface
15	Handsheets from 80:20 blend of Medium "C" and fresh kraft	None	9.3	347	2374	3000 +	77	52.8	At liner-adhesive interface
16	Handsheets from 80:20 blend of Medium "C" and fresh kraft	Triton X-100, 0.1	9.5	--	--	--	22	56.7	Within medium

[a]Pin adhesion values for smooth side.
Note: The corrugator was operated at 150 f.p.m.

A fourth and final series of tests utilized a commercial semichemical medium pulp which was provided in a dewatered condition. Handsheets were prepared from the pulp at two wet pressures (50 and 75 lb.) with and without sizing agent. However, the resulting sheets proved to be excessively bulky and rough and failure in the pin adhesion test occurred primarily within the experimental medium.

On the basis of the exploratory tests the decision was made to use commercial Mediums "B" and "C" with suitable surface modifications to provide the desired range in wettability. It was also decided to include handsheets prepared from Medium "C" and fresh kraft. Two of the aforementioned series would provide for comparison of relatively smooth and rough surfaces and all three would provide a range in surface receptivity. In addition, a range in Bendtsen porosity from approximately 400 to 1800 ml./min. would be provided considering the three units.

PREPARATION OF MEDIUM FOR CORRUGATING

A large number of sheets from each of the selected three units was prepared to provide an adequate supply for physical tests and for corrugating at four speeds with two adhesives. The first unit was comprised of commercial Medium "B" as is and after surface treating with 0.01, 0.02, and 0.05% of Aquapel 364 in ether solution. The second unit was comprised of commercial medium "C" as is and after surface treating with 0.01% of Triton X-100 and 0.011 and 0.05% of Aquapel 364. Both materials were applied from ether solution. The third unit was comprised of handsheets formed at pH 7.5-8.0 from a 70:30 blend of Medium "C" and fresh long-fiber kraft. The pulps were beaten separately and then combined to provide a freeness of 460 cc. C.S.F. Handsheets equivalent to 26-lb. medium were prepared without additives and with 0.04 and 0.1% of Aquapel 360X plus equal amounts of Kymene 557. The sheets were wet pressed one minute between blotters and then four minutes between a polished plate and blotters at 75-lb. pressure. One-half of the unsized handsheets were surface treated with 0.01% of Triton X-100 to improve receptivity. For purposes of control, the "untreated" commercial mediums were sprayed with ether. Sheets processed in the manner described were tested for basis weight, caliper, apparent density, water drop, contact angle against starch and distilled water, Bendtsen smoothness and porosity, and I.G.T. surface bonding strength. Results are recorded in Table IV.

CORRUGATOR TRIALS

Corrugator trials were subsequently carried out utilizing the aforementioned mediums and two adhesives. The experimental mediums were attached to a 4-mil kraft carrier sheet and the corrugator was operated at speeds of 150, 300, 450, and 600 ft./min. using a transfer-doctor roll clearance of 0.008 in. The slightly reduced glue line was utilized in an effort to emphasize differences in adhesional properties. The adhesives used were a conventional two-step starch adhesive prepared according to the Stein-Hall procedure previously utilized on this project (1, 2), and a one-step adhesive prepared according to a modified Corn Products procedure (4) as follows:

A starch suspension comprised of 10.15 lb. of Bondcor C (airdry basis) in 14,920 ml. of tap water was stirred in a 45-liter stainless steel drum contained in a 100-liter water bath which was adjusted to 115°F. When the temperature of the starch suspension reached 115°F., a caustic solution comprised of 142.3 g. of sodium hydroxide dissolved in 3910 ml. of tap water at 130°F. was added with stirring over a period of 4-5 minutes. The temperature of the suspension was maintained at 115°F. until the viscosity reached 25 seconds as measured by the

TABLE IV

PROPERTIES OF EXPERIMENTAL CORRUGATING MEDIUM

Corrugating Medium Sample No.	Description	Basis Wt., lb./1000 ft.²	Caliper, pt.	Apparent Density	Water Drop, sec.		Contact Angle, θ, degrees				Bendtsen Smoothness, ml./min.		Bendtsen Porosity, ml./min.	I.C.T. Bending Strength, kp.-cm./sec. in-direction	
							Against Water		Against Starch[b]						
					Rough Side	Smooth Side	Rough Side	Smooth Side	Rough Side	Smooth Side	Rough Side	Smooth Side		Rough Side	Smooth Side
B-1	Commercial Medium "B" as is	26.9	9.9	2.7	26	31	--[c]	--[c]	73	79	2472	2082	1662	81	112
B-2	Commercial Medium "B" surface treated with 0.01% Aquapel 364	26.5	10.0	2.6	187	254	95	100	100	104	2483	2194	1803	99	128
B-3	Commercial Medium "B" surface treated with 0.02% Aquapel 364	26.3	10.0	2.6	558	551	95	100	102	105	2532	2136	1710	108	153
B-4	Commercial Medium "B" surface treated with 0.05% Aquapel 364	26.4	9.9	2.7	600 +	600 +	113	113	123	124	2460	2217	1840	97	127
C-1	Commercial Medium "C" surface treated with 0.01% Triton X-100	25.5	10.7	2.4	35	--	--[c]	--	61	--	3000 +	--	880	179	--
C-2	Commercial Medium "C" as is	25.1	10.2	2.5	63	--	--[c]	--	89	--	3000 +	--	854	164	--
C-3	Commercial Medium "C" surface treated with 0.011% Aquapel 364	25.3	10.6	2.4	412	--	104	--	100	--	3000 +	--	882	172	--
C-4	Commercial Medium "C" surface treated with 0.05% Aquapel 364	25.5	10.6	2.4	600 +	--	110	--	124	--	3000 +	--	866	180	--
H-1	Handsheets[a] - surface treated with 0.01% Triton X-100	25.4	11.3	2.2	34	33	--[c]	--[c]	57	55	3000 +	2463	563	111	153
H-2	Handsheets[a] - as is	25.7	10.9	2.4	57	53	--[c]	--[c]	76	74	3000 +	2431	479	117	114
H-3	Handsheets[a] - sized with 0.04% Aquapel 360X	26.1	10.6	2.5	204	225	52	72	90	91	3000 +	2295	426	108	159
H-4	Handsheets[a] - sized with 0.1% Aquapel 360X	26.0	11.0	2.4	600 +	600 +	106	104	104	104	3000 +	2441	461	103	142

[a]Handsheets formed from 70:30 blend of Medium "C" and fresh kraft.
[b]Conventional two-step corrugating starch.
[c]Penetrated too rapidly for measurements to be made.

Stein-Hall flow viscometer. At this point, 20.93 g. of alum dissolved in 125.6 ml. of room-temperature tap water was added and stirred in for two minutes. Finally, 132 g. of borax was added with stirring.

The aforementioned procedure is essentially the same as that described in Progress Report Three; however, it was found in the present case that the time required to reach the required Stein-Hall flow viscosity was 30 minutes compared to 20 minutes in the previous work. Slight differences were also noted in the solids content and pH but perhaps more significant was a change in the gel point. The adhesive used in the current work had a gel point of 150°F. compared to 145-146° in the earlier work (3). The relatively high gel point for the most recent batch was subsequently thought to influence adhesion at high corrugating speed. Inspection of samples immediately after corrugation at 600 f.p.m. indicated that the one-step adhesive had not set-up adequately. While the one-step procedure produced a more uniformly swollen starch suspension than the conventional procedure, some variation in particle size persisted.

Pin adhesion results for the experimental medium are recorded in Table V. The effect of corrugating speed on pin adhesion at the various receptivities is shown graphically in Fig. 1-10. Pin adhesion as a function of receptivity (water drop value) under conditions of optimum surface geometry (smoothness) is presented in Fig. 11-16. Results obtained at 600 f.p.m. were not included in Fig. 12, 14, and 16 because of the indicated failure of the one-step adhesive to set-up adequately under this condition. In cases where pin adhesion is plotted as a function of corrugating speed, the relationship for the one-step adhesive beyond 450 f.p.m. is of questionable significance and is, therefore, segmented. The apparent optimum water drop values for the three mediums plotted as a function of the Bendtsen porosity are presented in Fig. 17. The selection of points for this relationship

TABLE V

PIN ADHESION RESULTS FOR EXPERIMENTAL MEDIUM

Sample No.	Side	Corrugating Speed, f.p.m.	Pin Adhesion: Using Conventional Starch Adhesive	Pin Adhesion: Using One-Step Starch Adhesive
B-1	Smooth	150	60.9	65.8
		300	58.5	62.6
		450	58.4	57.0
		600	50.3	44.8[b]
	Rough	150	56.1	63.3
		300	51.2	58.4
		450	45.8	55.1
		600	39.9	32.0[b]
B-2	Smooth	150	60.4	65.0
		300	60.7	65.4
		450	58.4	63.2
		600	54.4	45.3[b]
	Rough	150	56.2	61.9
		300	57.5	59.1
		450	50.1	61.5
		600	45.8	44.7[b]
B-3	Smooth	150	56.4	63.2
		300	56.3	61.4
		450	53.6	62.6
		600	45.0	36.9[b]
	Rough	150	56.9	57.4
		300	52.7	56.4
		450	47.0	52.5
		600	41.4	38.4[b]
B-4	Smooth	150	51.4	55.0
		300	51.4	59.0
		450	45.4	55.4
		600	37.8	38.1[b]
	Rough	150	44.1	49.5
		300	39.4	48.1
		450	41.7	50.8
		600	34.0	33.9[b]
C-1	Rough[a]	150	71.4	75.9
		300	68.1	77.7
		450	67.7	69.5
		600	52.2	56.9[b]
C-2	Rough[a]	150	74.6	81.9
		300	72.0	77.8
		450	65.2	78.5
		600	63.2	58.1[b]
C-3	Rough[a]	150	67.4	76.6
		300	65.1	77.3
		450	61.1	69.0
		600	51.7	47.1[b]

See end of table for footnotes.

TABLE V (Continued)

PIN ADHESION RESULTS FOR EXPERIMENTAL MEDIUM

Sample No.	Side	Corrugating Speed, f.p.m.	Pin Adhesion: Using Conventional Starch Adhesive	Pin Adhesion: Using One-Step Starch Adhesive
C-4	Rough[a]	150	54.4	61.0
		300	52.0	57.7
		450	52.3	55.5
		600	52.7	51.9[b]
H-1	Smooth	150	68.2	75.6
		300	65.7	71.8
		450	59.2	70.0
		600	62.0	50.7[b]
	Rough	150	71.4	77.6
		300	67.4	68.6
		450	64.4	69.9
		600	58.2	45.9[b]
H-2	Smooth	150	68.3	72.0
		300	64.4	77.0
		450	61.4	72.5
		600	57.3	52.9[b]
	Rough	150	68.6	74.3
		300	63.1	75.7
		450	60.7	68.7
		600	58.3	45.8[b]
H-3	Smooth	150	64.1	66.4
		300	59.8	66.0
		450	59.3	63.4
		600	58.1	55.2[b]
	Rough	150	67.3	70.6
		300	66.7	67.3
		450	58.9	63.1
		600	50.9	48.0[b]
H-4	Smooth	150	54.3	57.9
		300	53.7	61.1
		450	54.2	58.6
		600	52.8	52.0[b]
	Rough	150	62.5	57.6
		300	62.8	62.9
		450	59.6	60.5
		600	49.7	48.8[b]

[a] Both sides of this medium had Bendtsen smoothness values of 3000 + ml./min.

[b] These values are considered low because of the indicated failure of the adhesive to set up adequately.

Note: Examination of the specimens from the pin adhesion tests indicated that, in general, the extent of failure at the medium-adhesive interface increased as corrugating speed and water resistance increased.

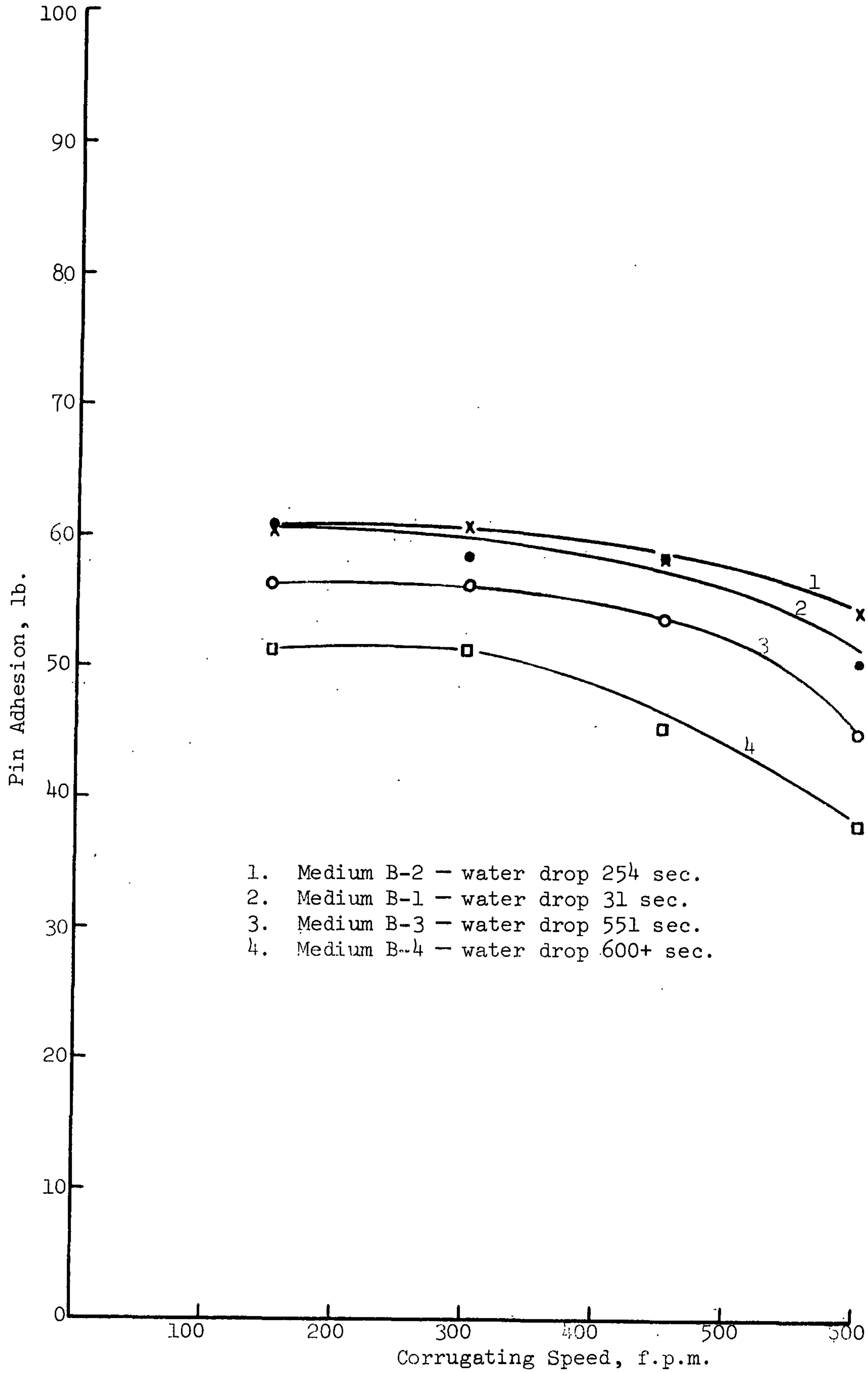

Figure 1. The Effects of Corrugating Speed and Surface Receptivity on Pin Adhesion for Medium "B" (Smooth Side) Using a Conventional Two-Step Starch Adhesive

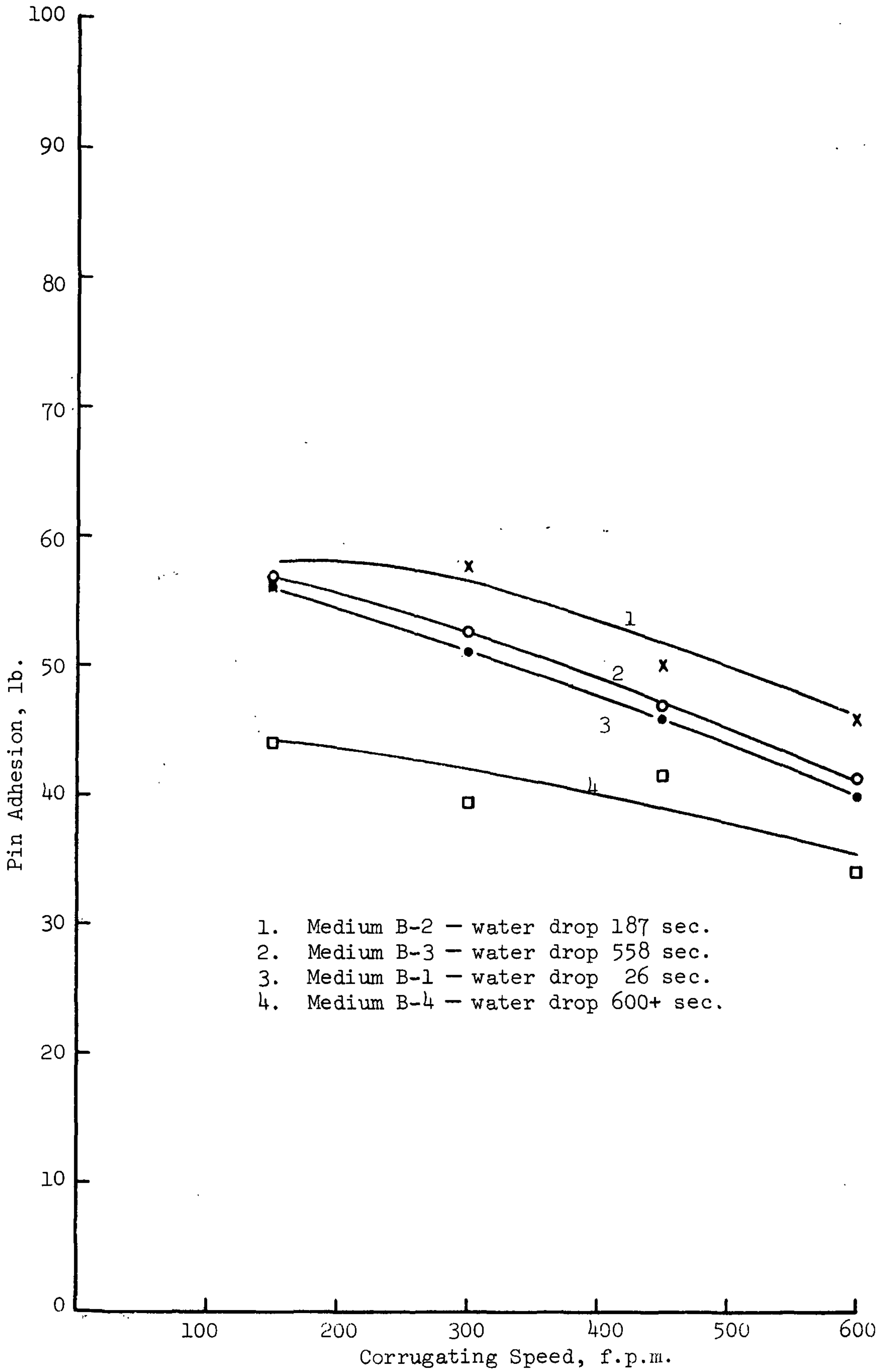

Figure 2. The Effects of Corrugating Speed and Surface Receptivity on Pin Adhesion for Medium "B" (Rough Side) Using a Conventional Two-Step Starch Adhesive

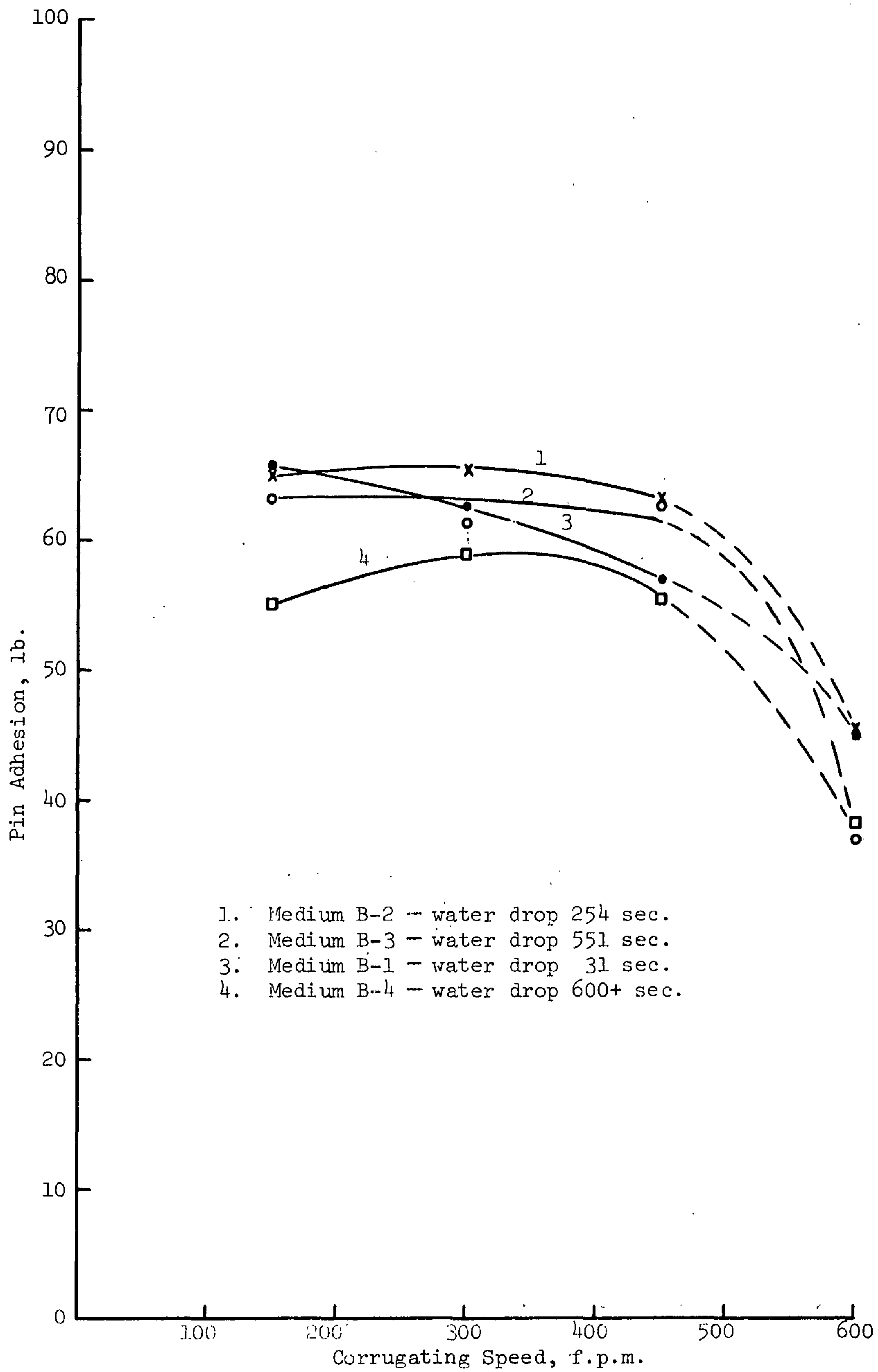

Figure 3. The Effects of Corrugating Speed and Surface Receptivity on Pin Adhesion for Medium "B" (Smooth Side) Using a One-Step Starch Adhesive

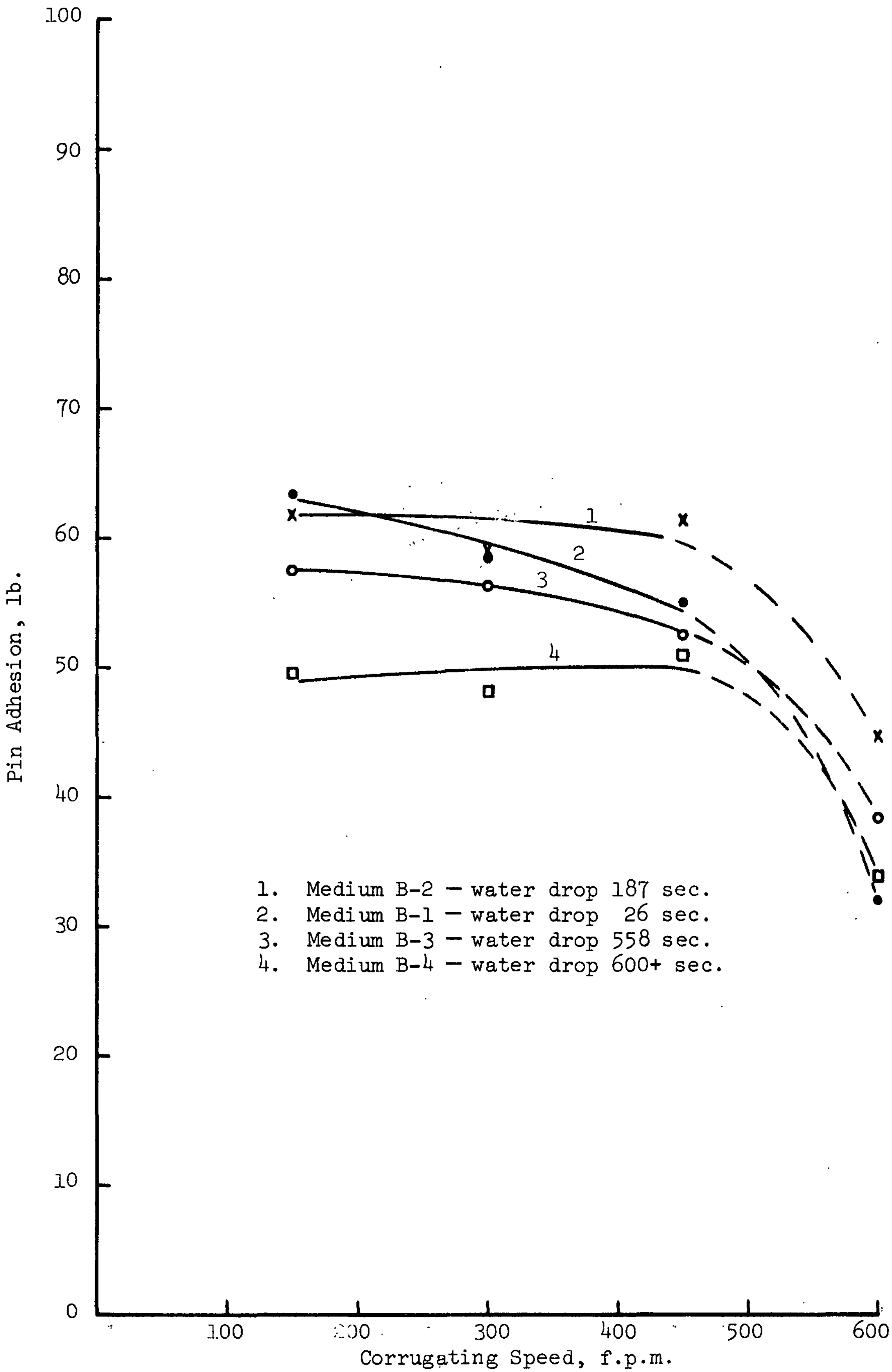

Figure 4. The Effects of Corrugating Speed and Surface Receptivity on Pin Adhesion for Medium "B" (Rough Side) Using a One-Step Starch Adhesive

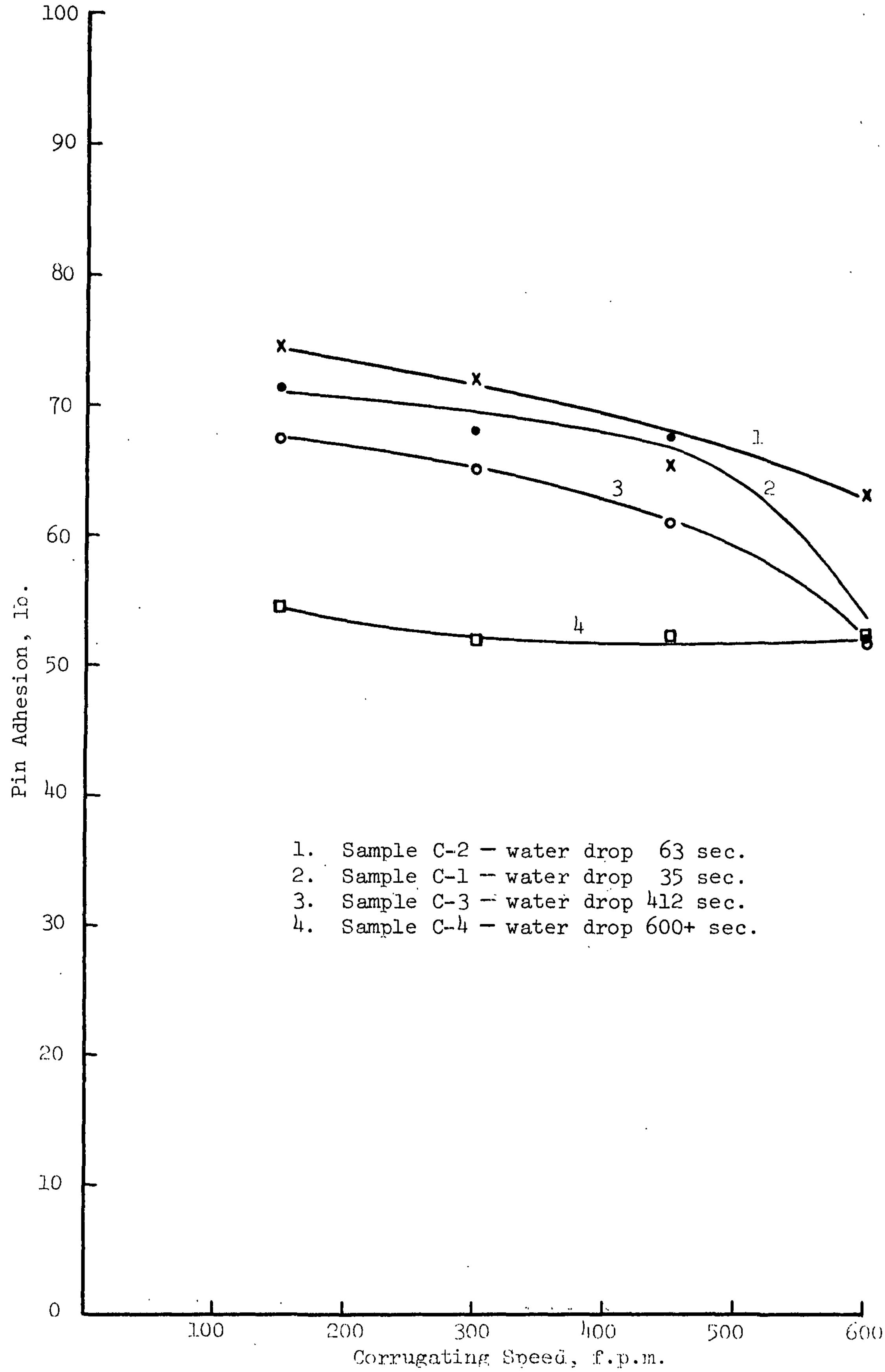

Figure 5. The Effects of Corrugating Speed and Surface Receptivity on Pin Adhesion for Medium "C" Using a Conventional Two-Step Starch Adhesive

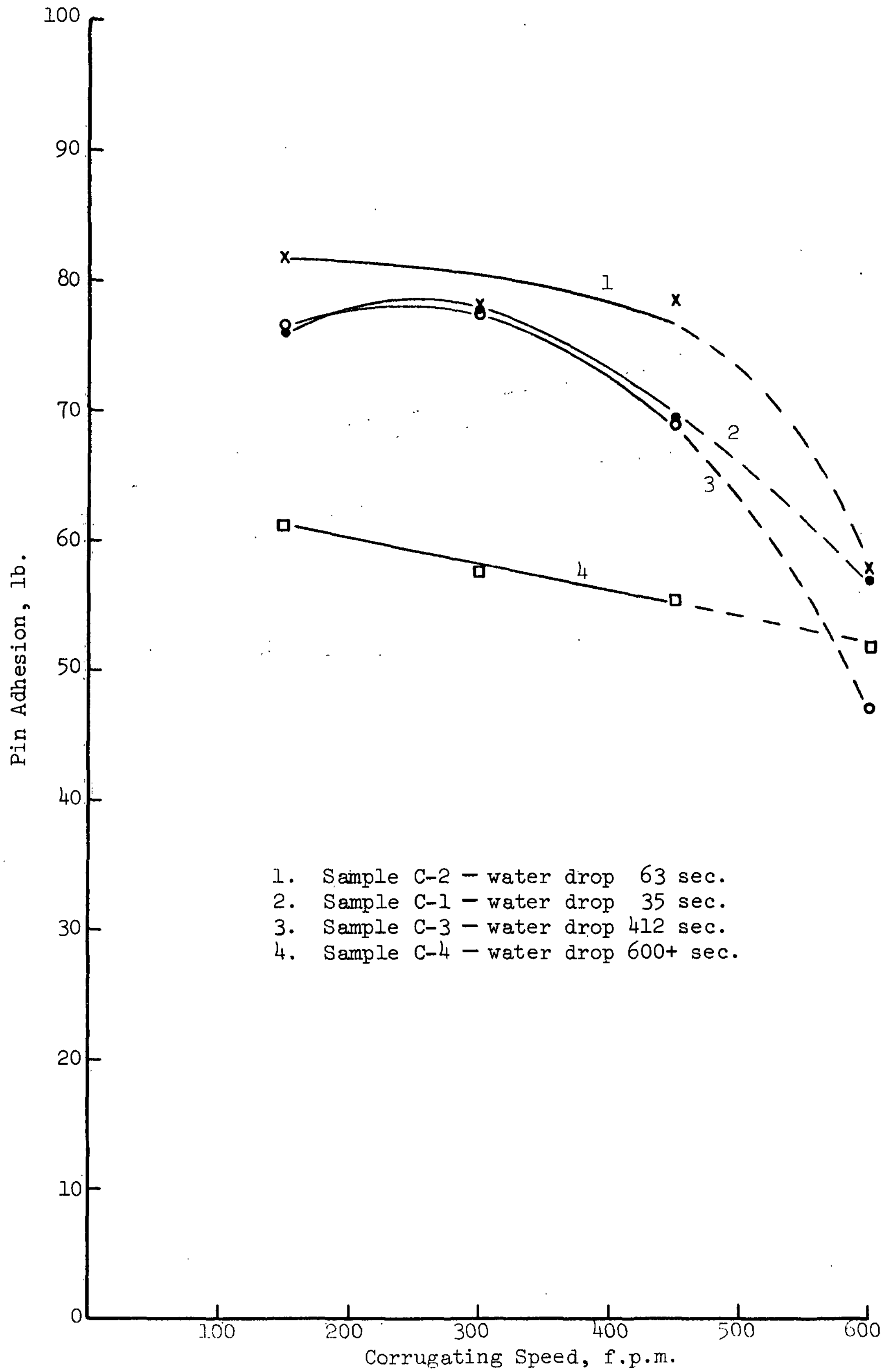

Figure 6. The Effects of Corrugating Speed and Surface Receptivity on Pin Adhesion for Medium "C" Using a One-Step Starch Adhesive

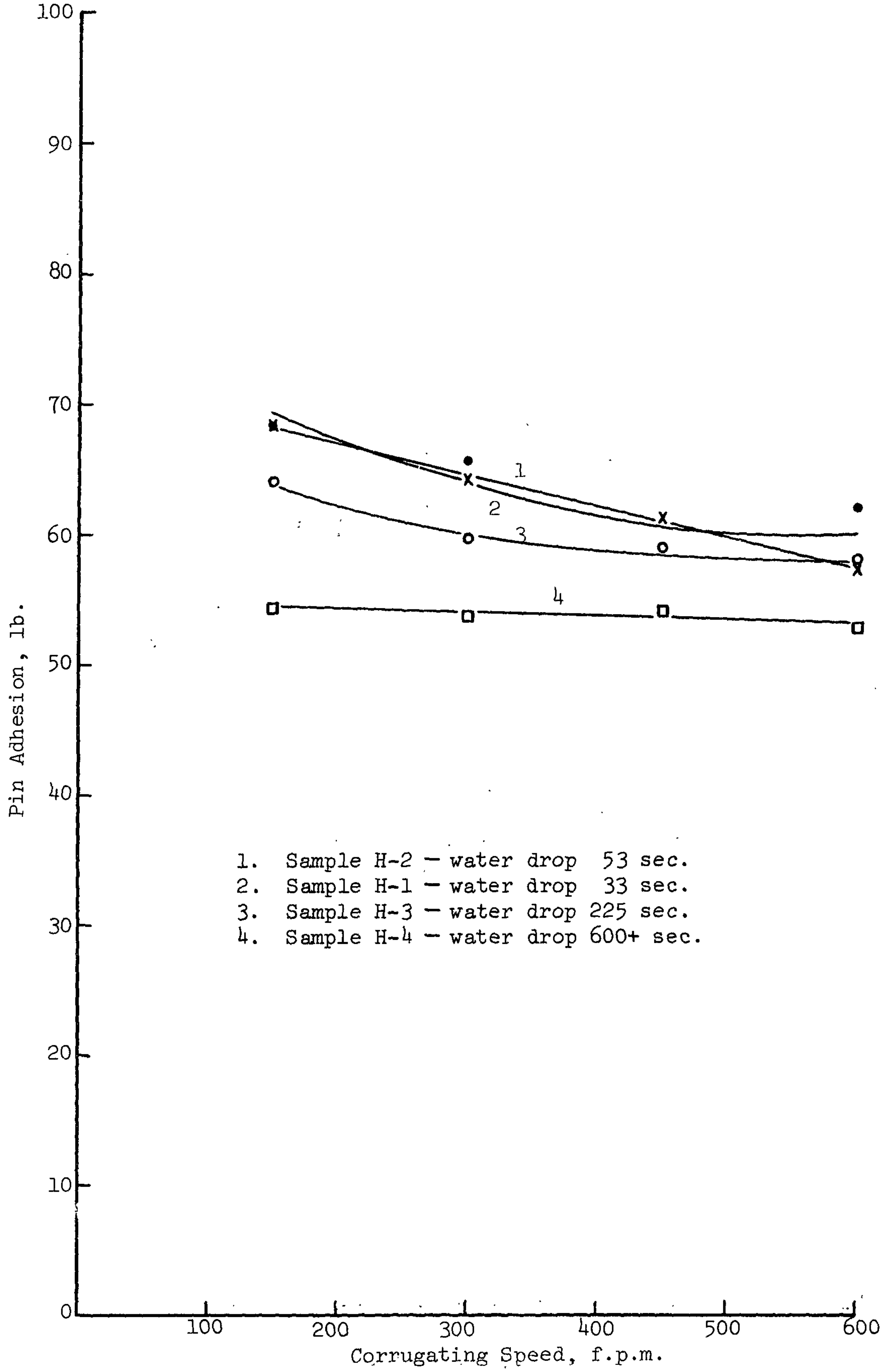

Figure 7. The Effects of Corrugating Speed and Surface Receptivity on Pin Adhesion for Medium "H" (Smooth Side) Using a Conventional Two-Step Starch Adhesive

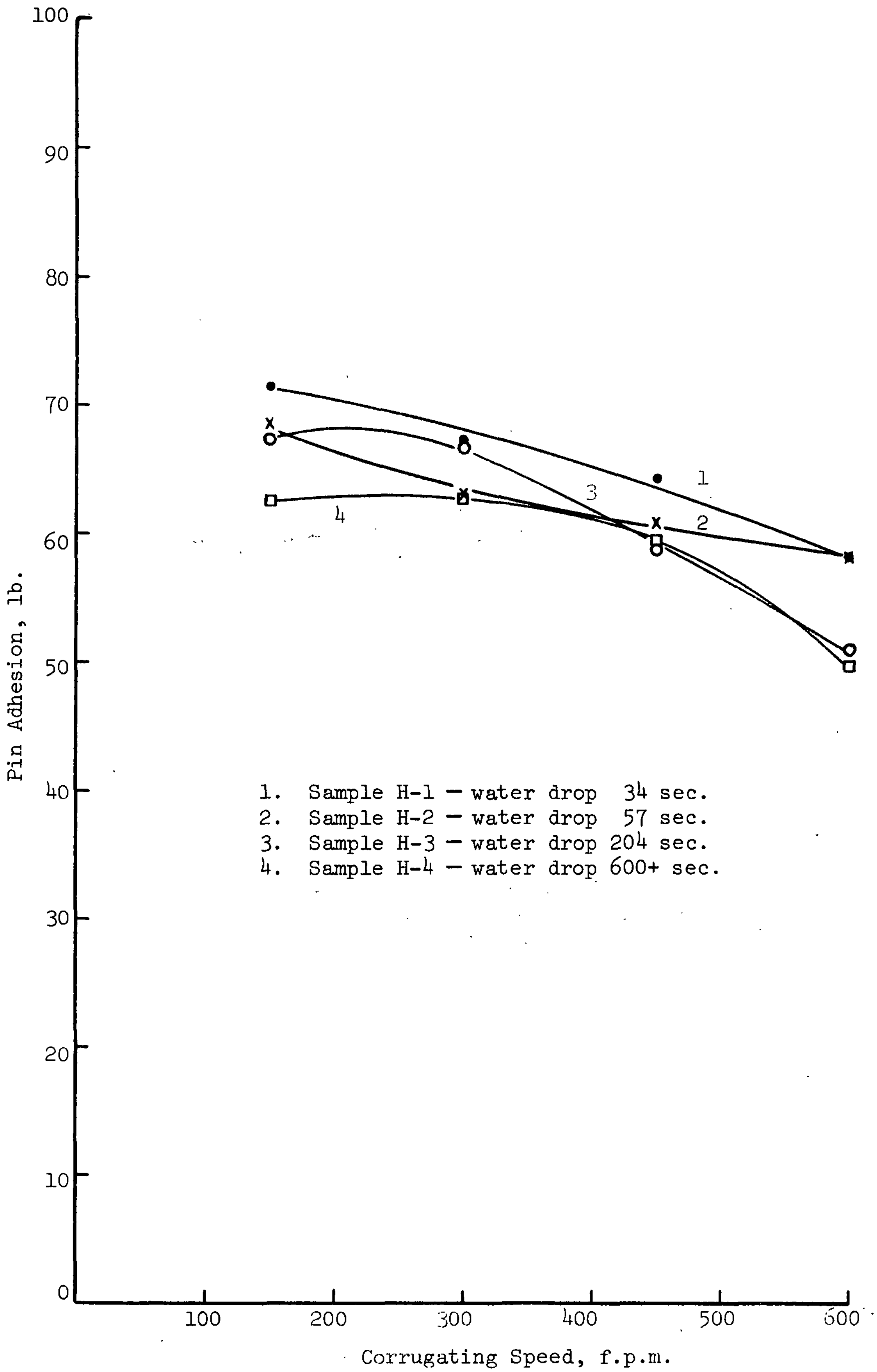

Figure 8. The Effects of Corrugating Speed and Surface Receptivity on Pin Adhesion for Medium "H" (Rough Side) Using a Conventional Two-Step Starch Adhesive.

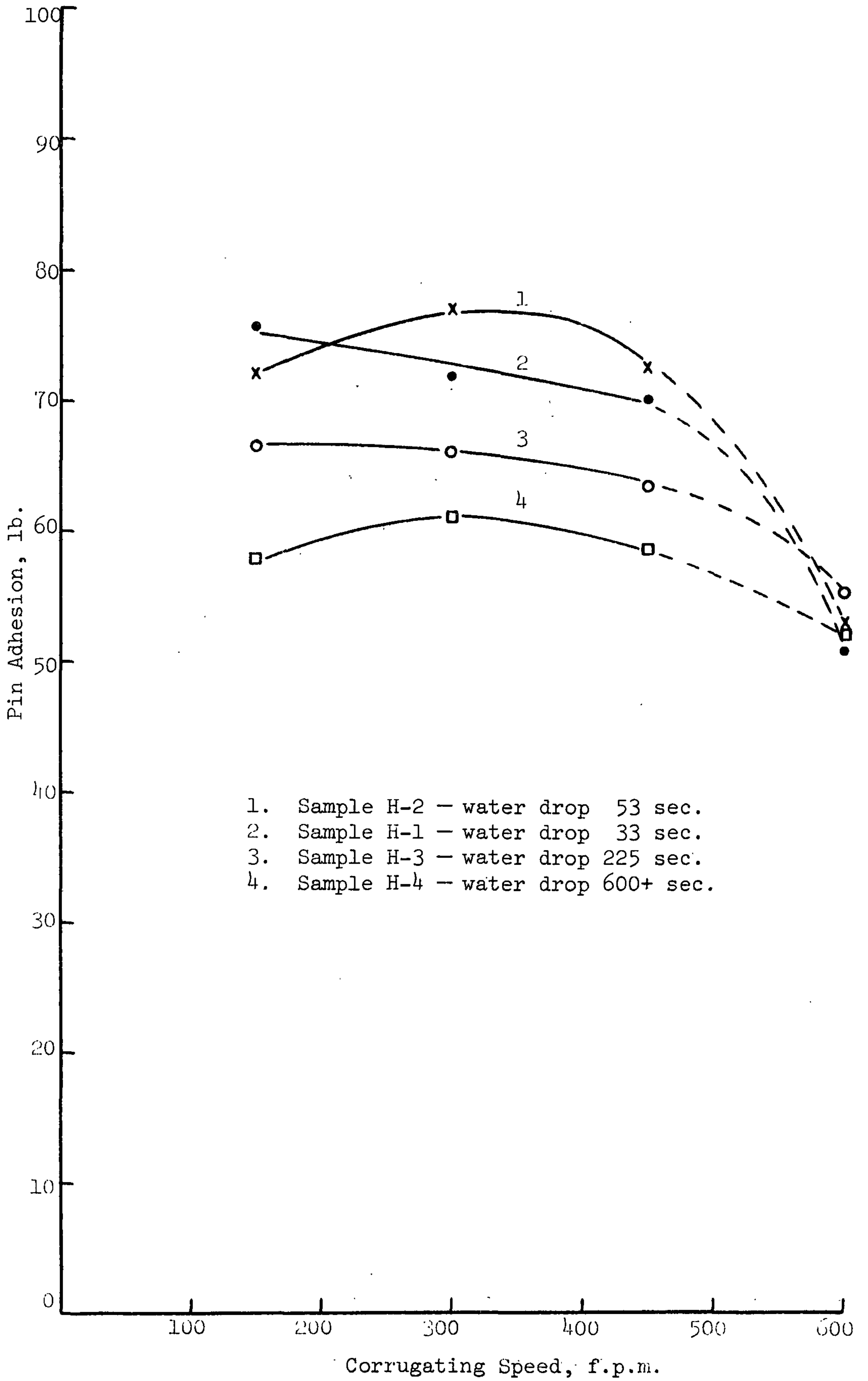

Figure 9. The Effects of Corrugating Speed and Surface Receptivity On Pin Adhesion for Medium "H" (Smooth Side) Using a One-Step Starch Adhesive

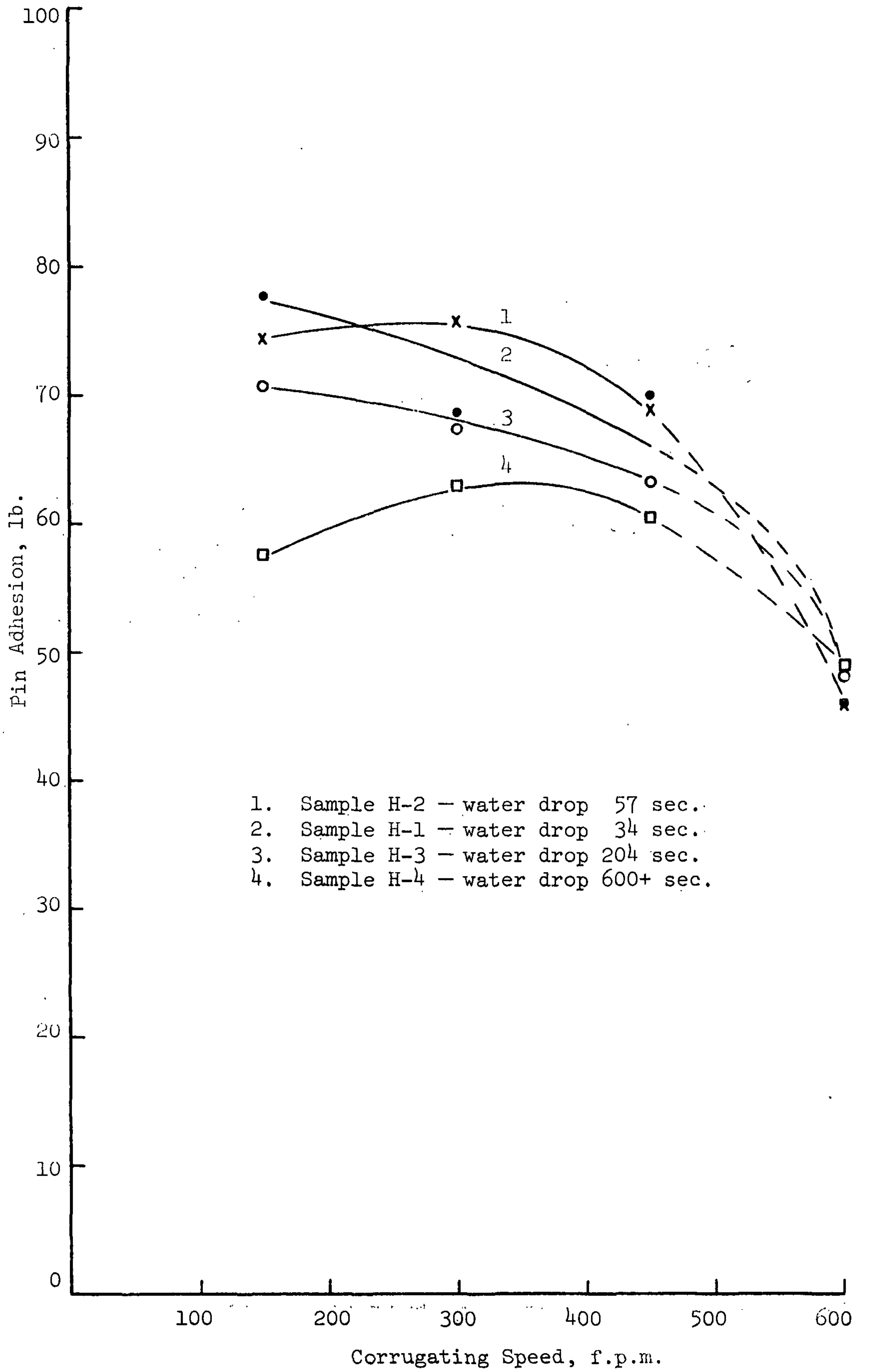

Figure 10. The Effects of Corrugating Speed and Surface Receptivity On Pin Adhesion for Medium "H" (Rough Side) Using a One-Step Starch Adhesive

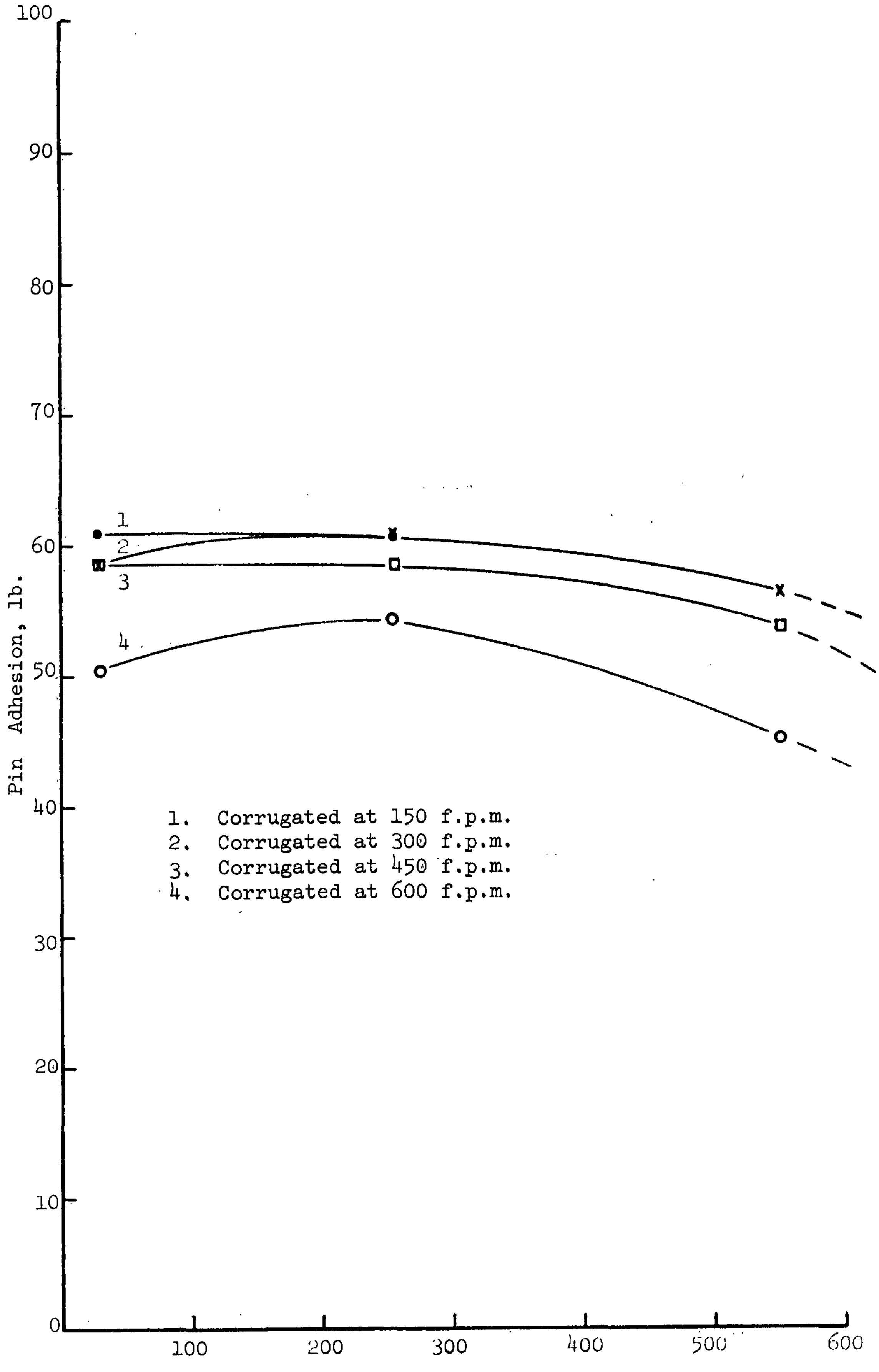

Figure 11. Pin Adhesion as a Function of Receptivity for Medium "B" (Smooth Side) Using a Conventional Two-Step Starch Adhesive

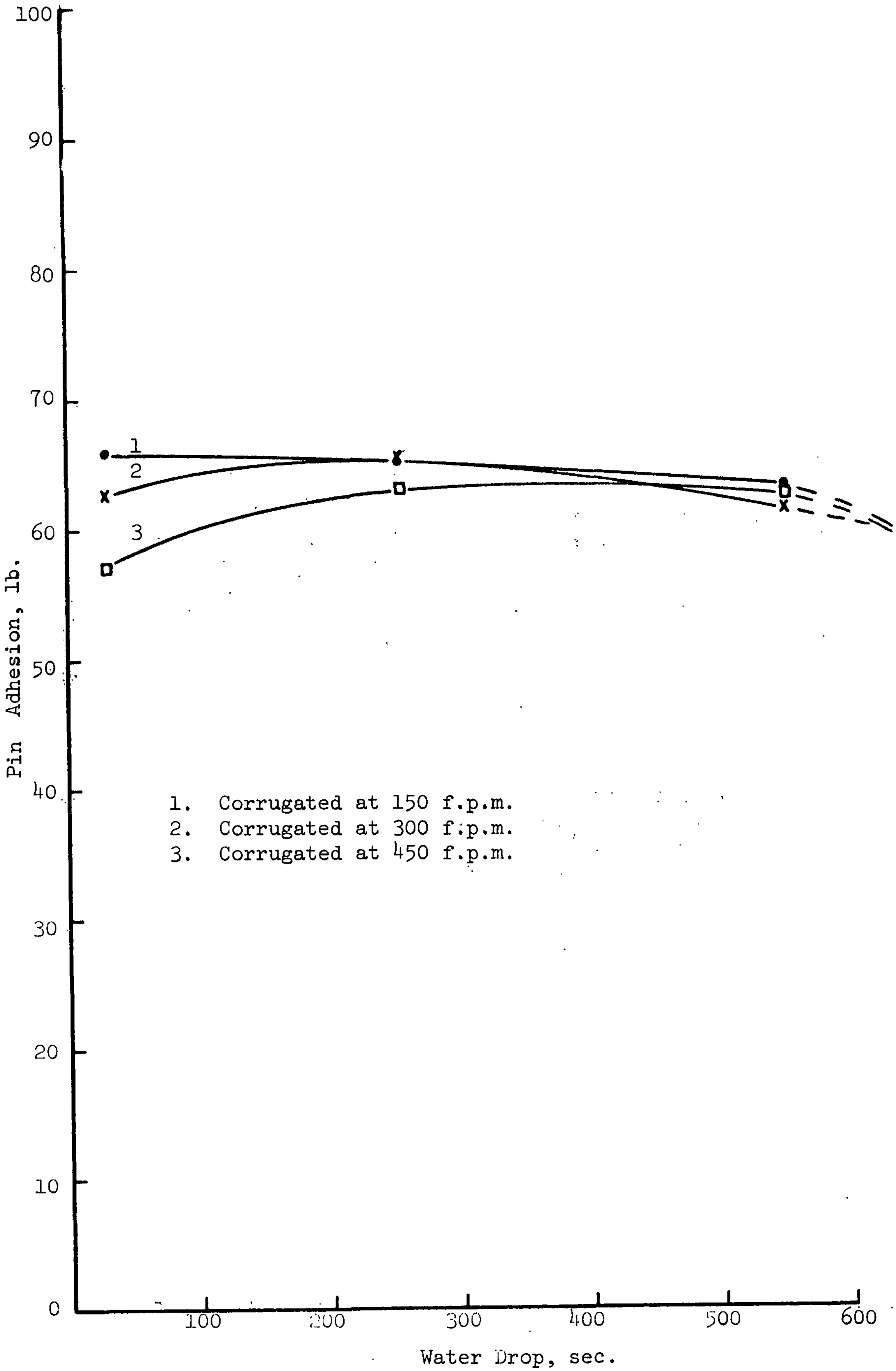

Figure 12. Pin Adhesion as a Function of Receptivity for Medium "B" (Smooth Side) Using a One-Step Starch Adhesive

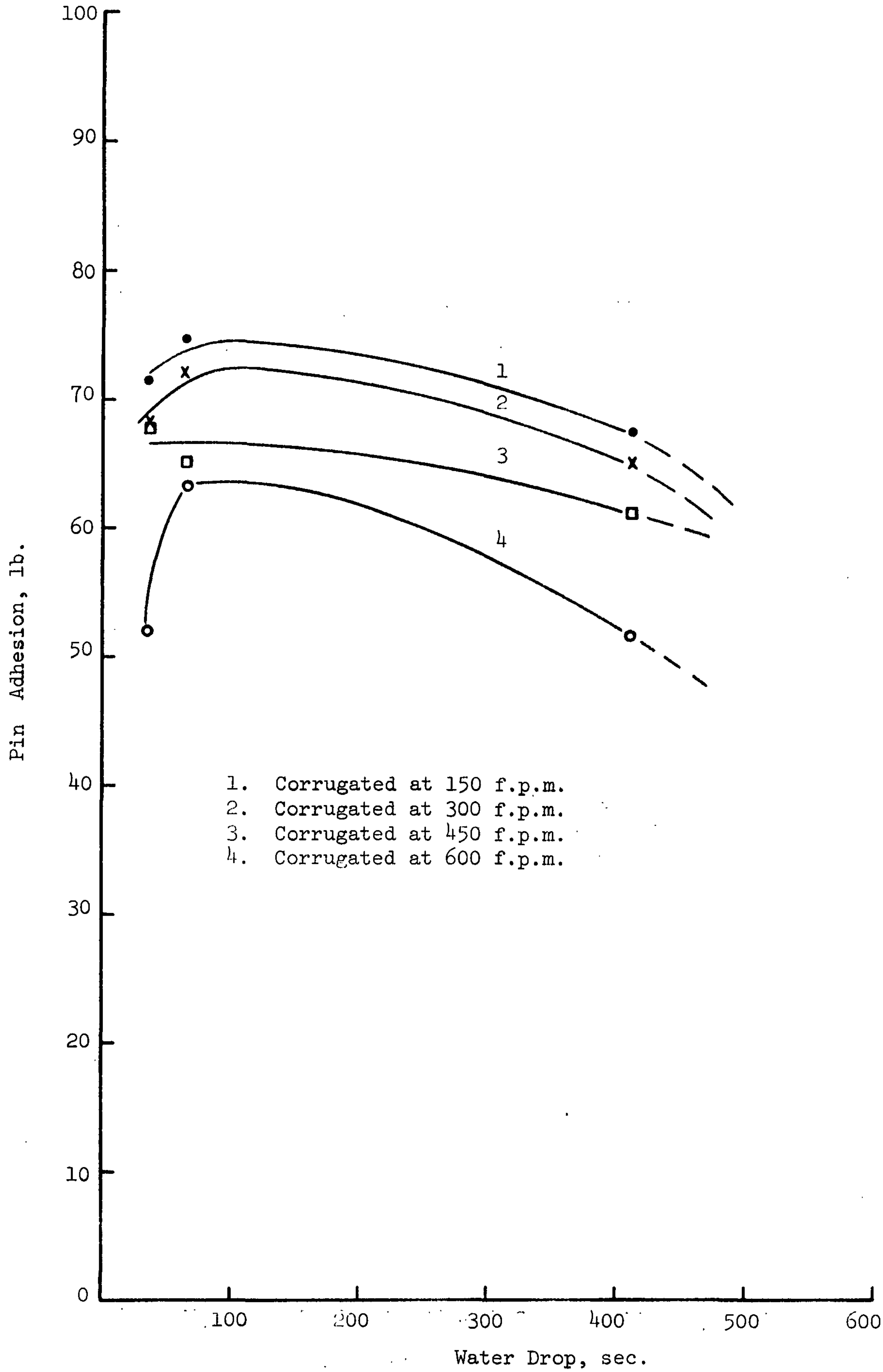

Figure 13. Pin Adhesion as a Function of Receptivity for Medium "C" Using a Conventional Two-Step Starch Adhesive

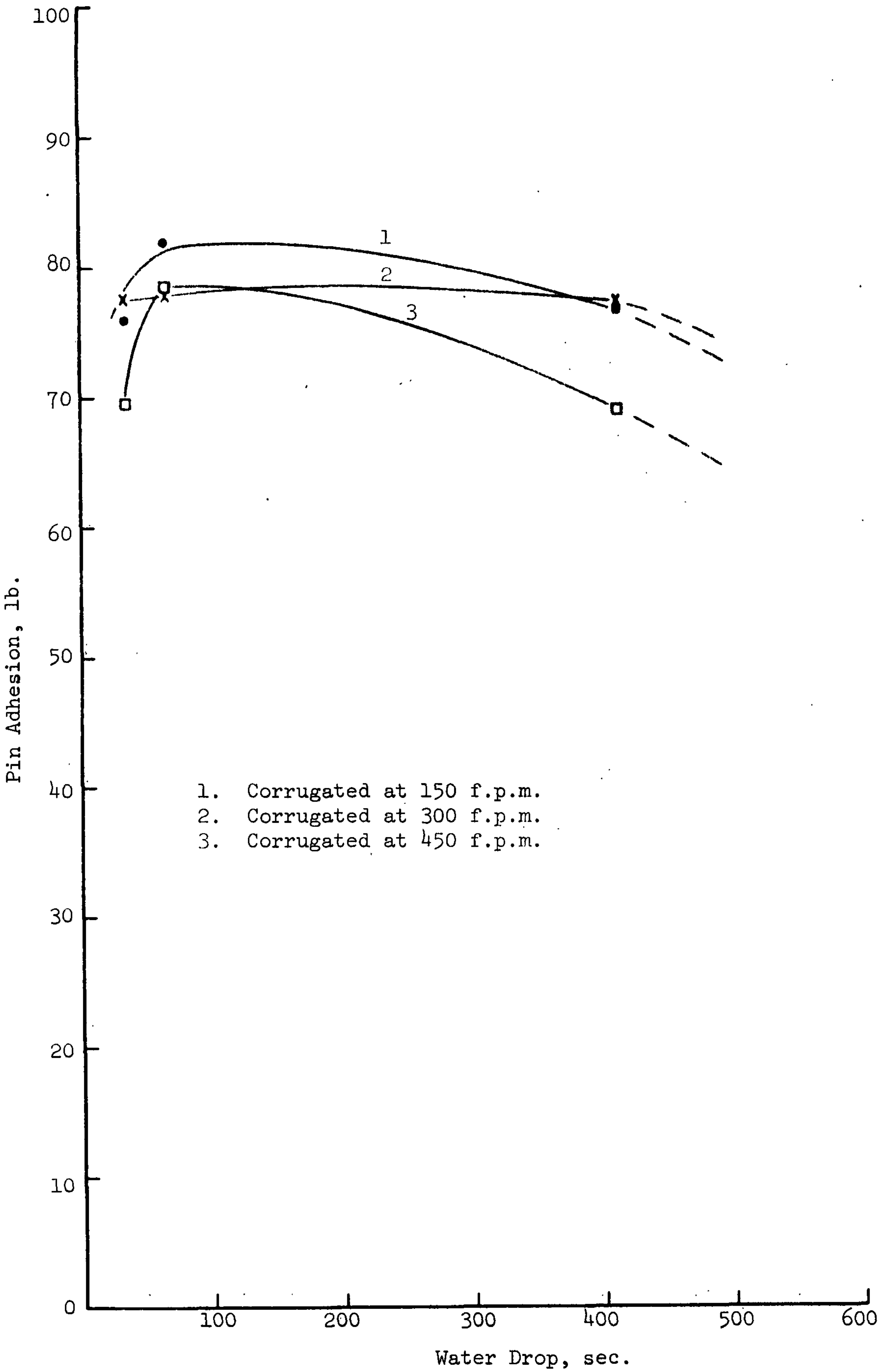

Figure 14. Pin Adhesion as a Function of Receptivity for Medium "C" Using a One-Step Starch Adhesive

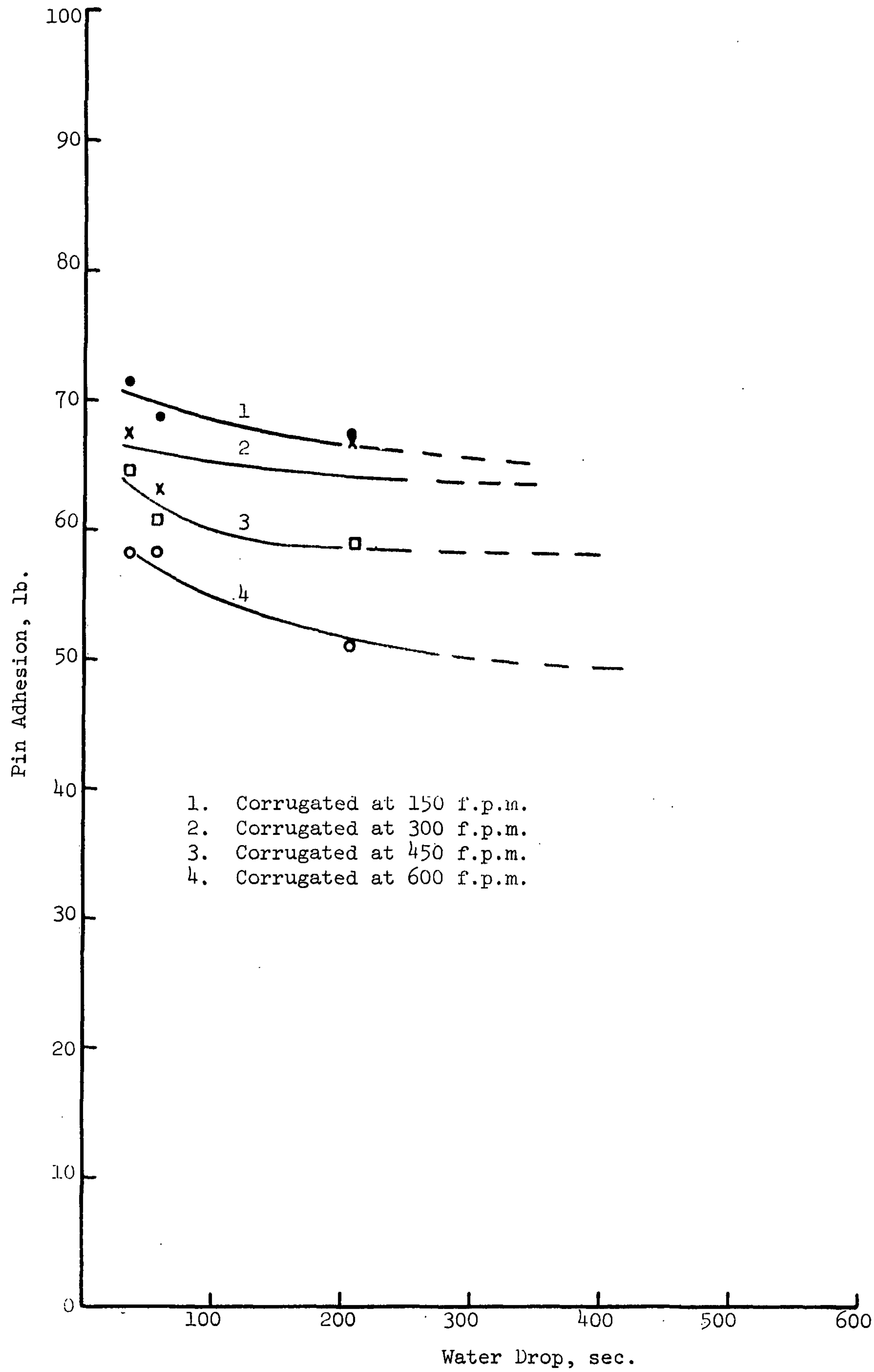

Figure 15. Pin Adhesion as a Function of Receptivity for Medium "H" (Rough Side) Using a Conventional Two-Step Starch Adhesive

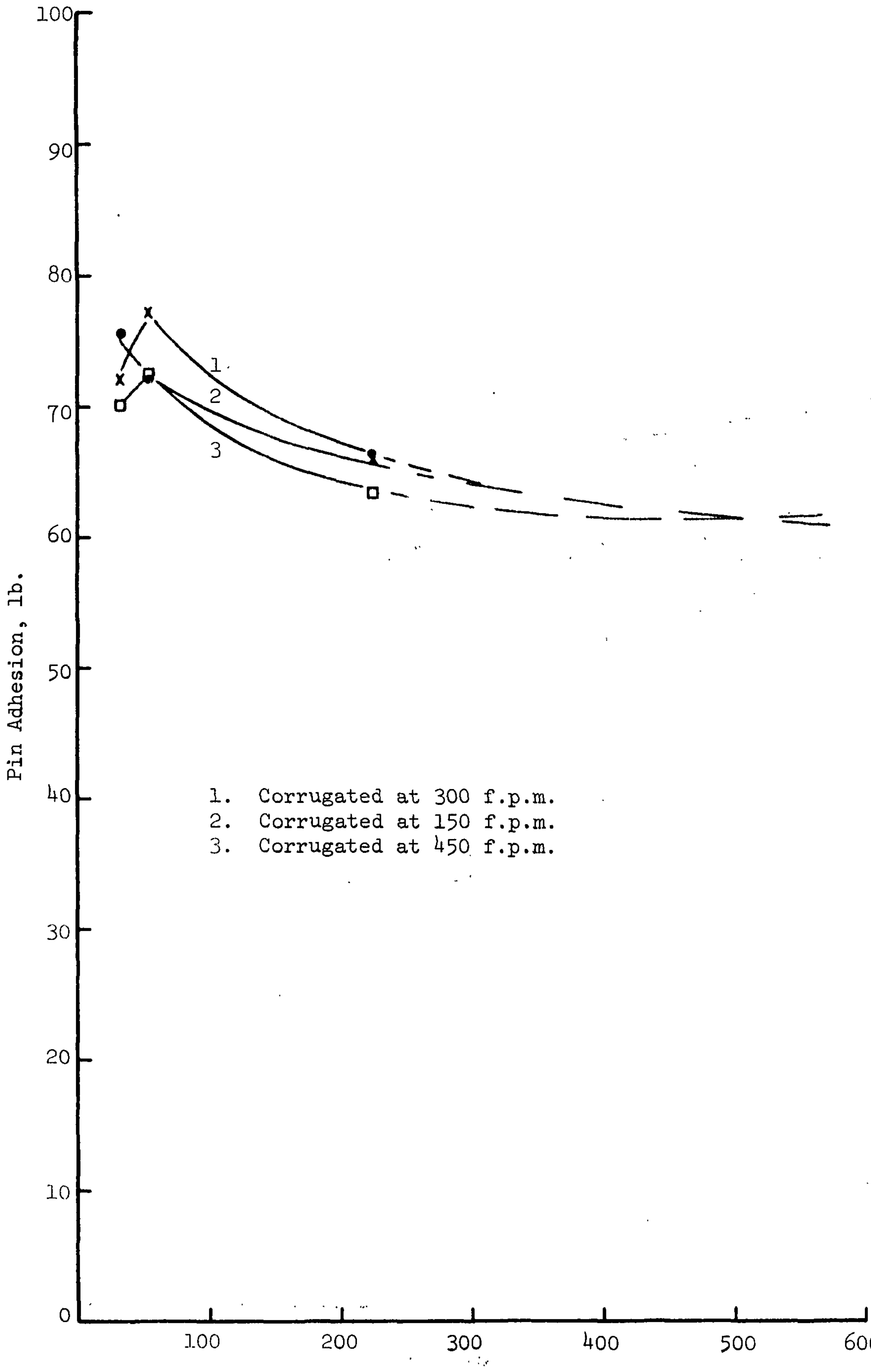

Figure 16. Pin Adhesion as a Function of Receptivity for Medium "H" (Smooth Side) Using a One-Step Starch Adhesive

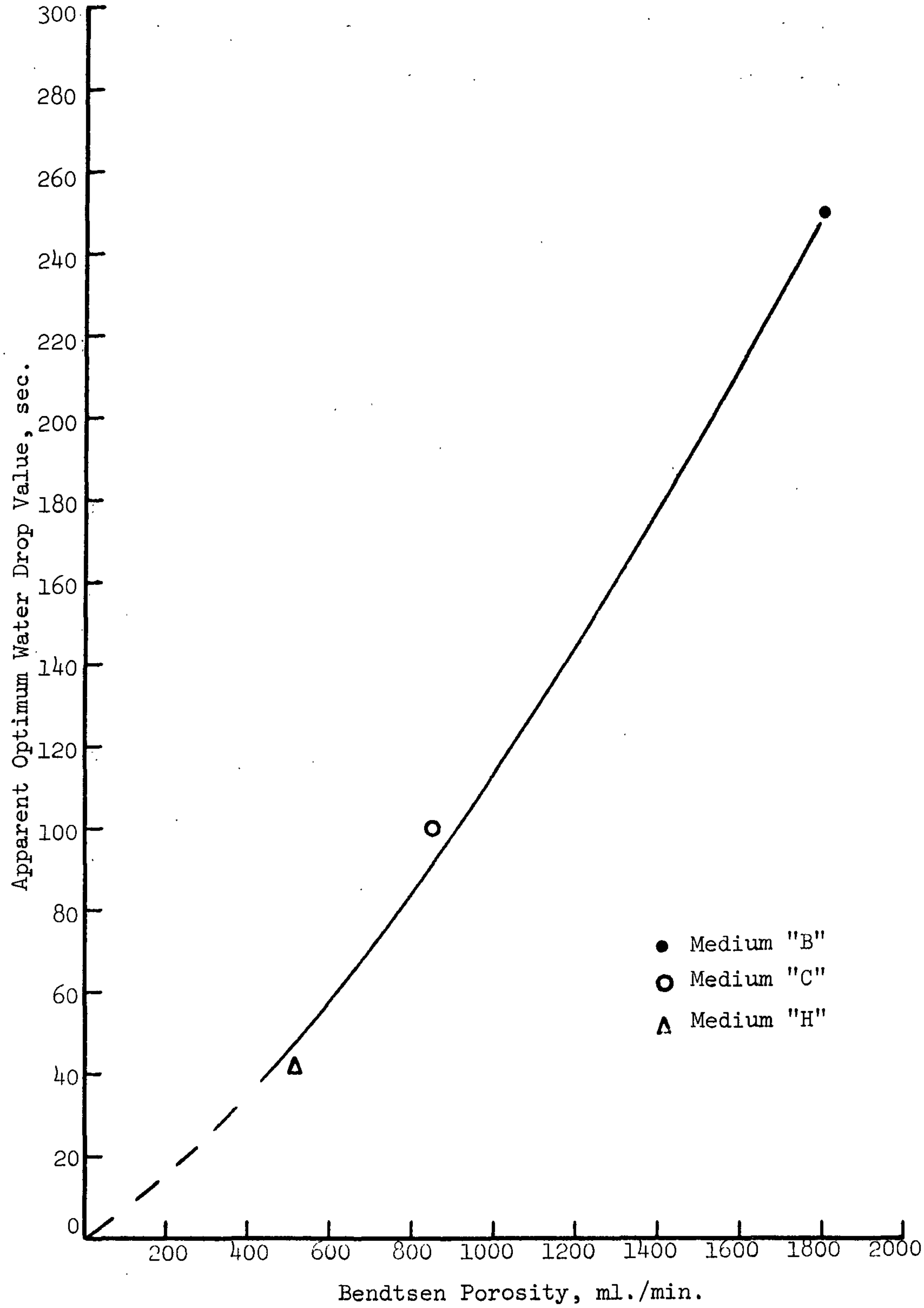

Figure 17. Apparent Optimum Receptivity (Water Drop) as a Function of Porosity

is described in the Discussion of Results section of this report. The effect of surface roughness on pin adhesion at optimum or near optimum receptivity is presented in Fig. 18 and 19 and comparisons of the two corrugating adhesives under optimum surface conditions for each are presented in Fig. 20 and 21.

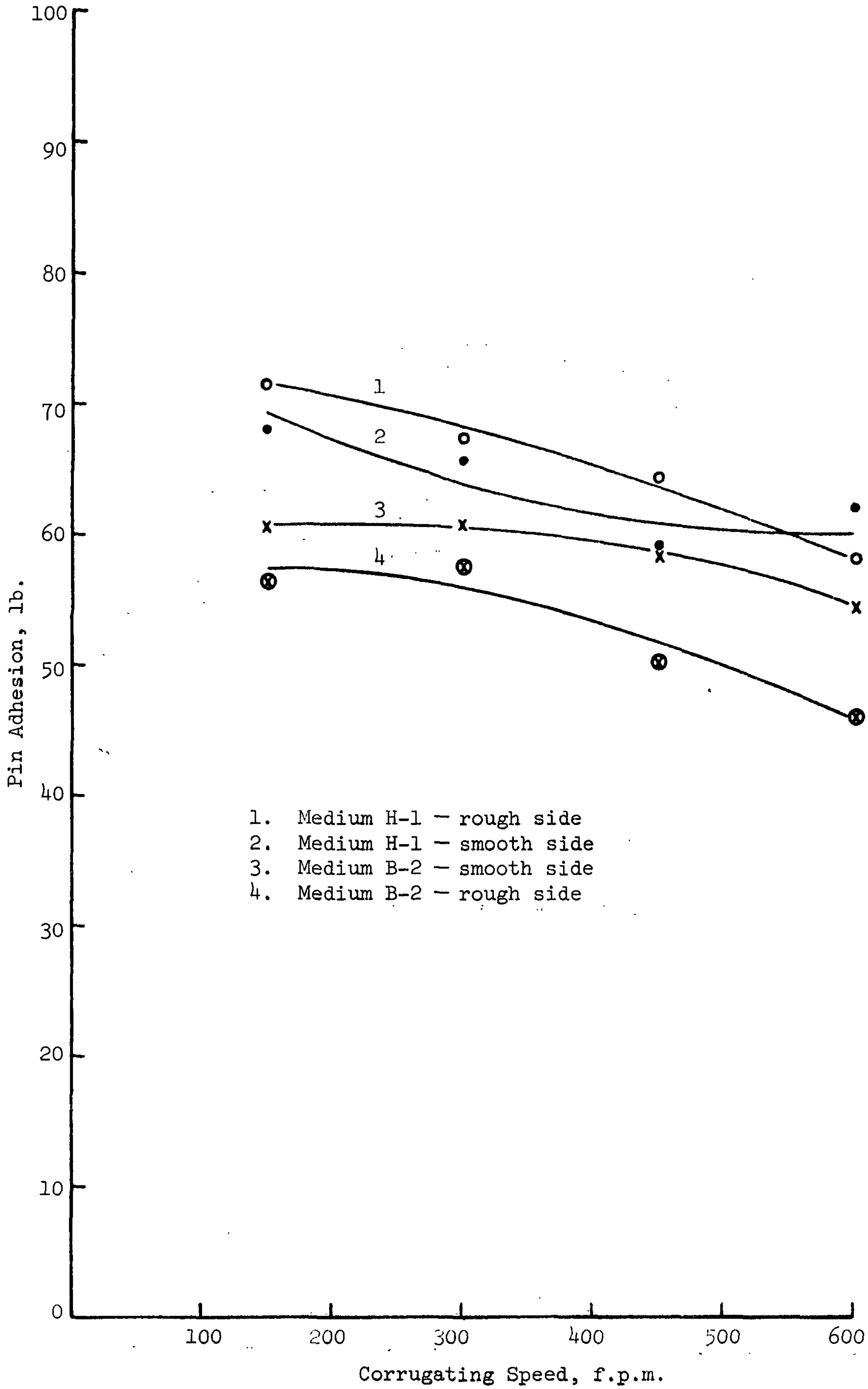

Figure 18. The Effect of Surface Roughness on Pin Adhesion Under Optimum Receptivity Conditions Using a Conventional Two-Step Starch Adhesive

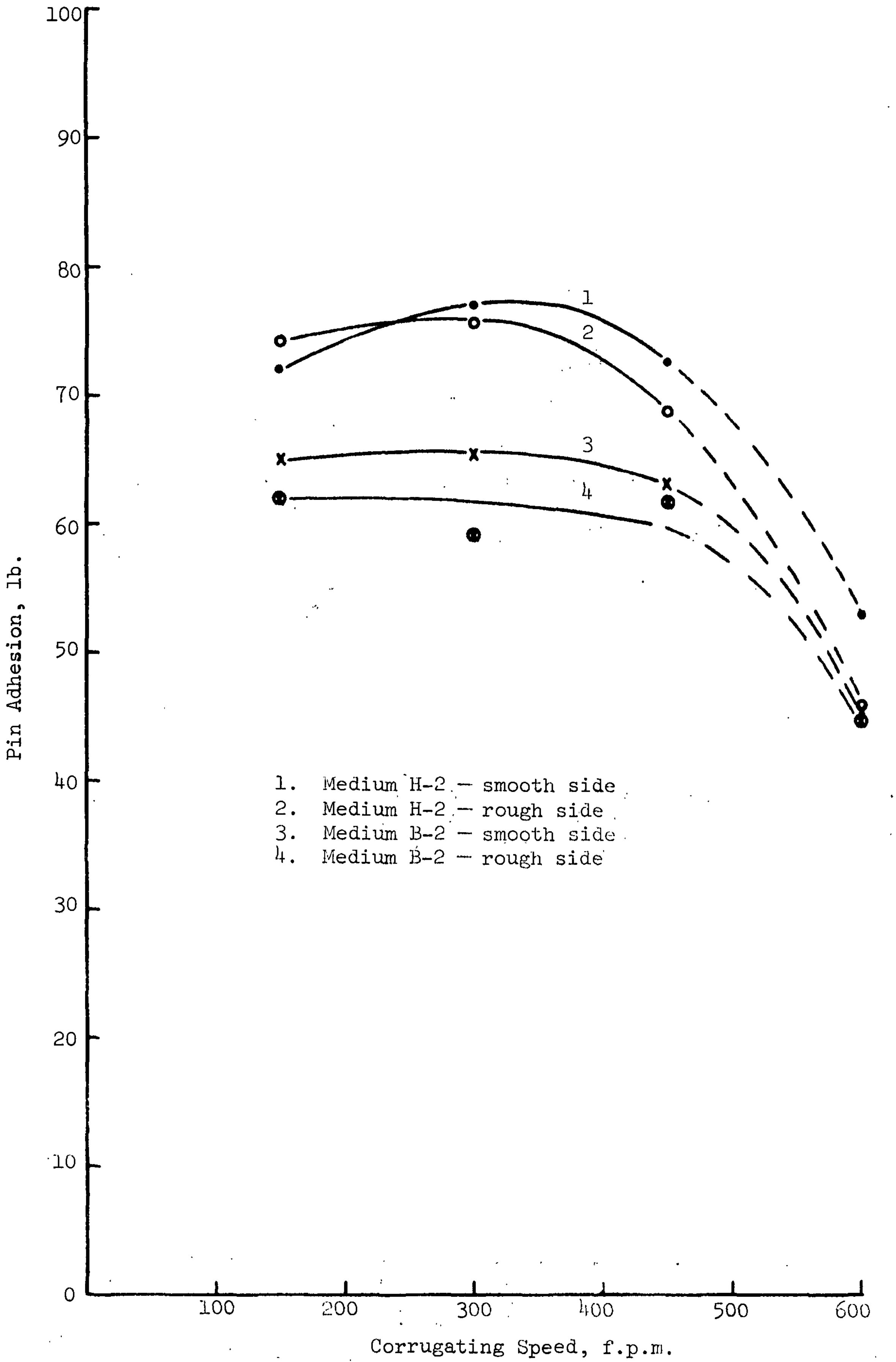

Figure 19. The Effect of Surface Roughness on Pin Adhesion Under Optimum Receptivity Conditions Using a One-Step Starch Adhesive

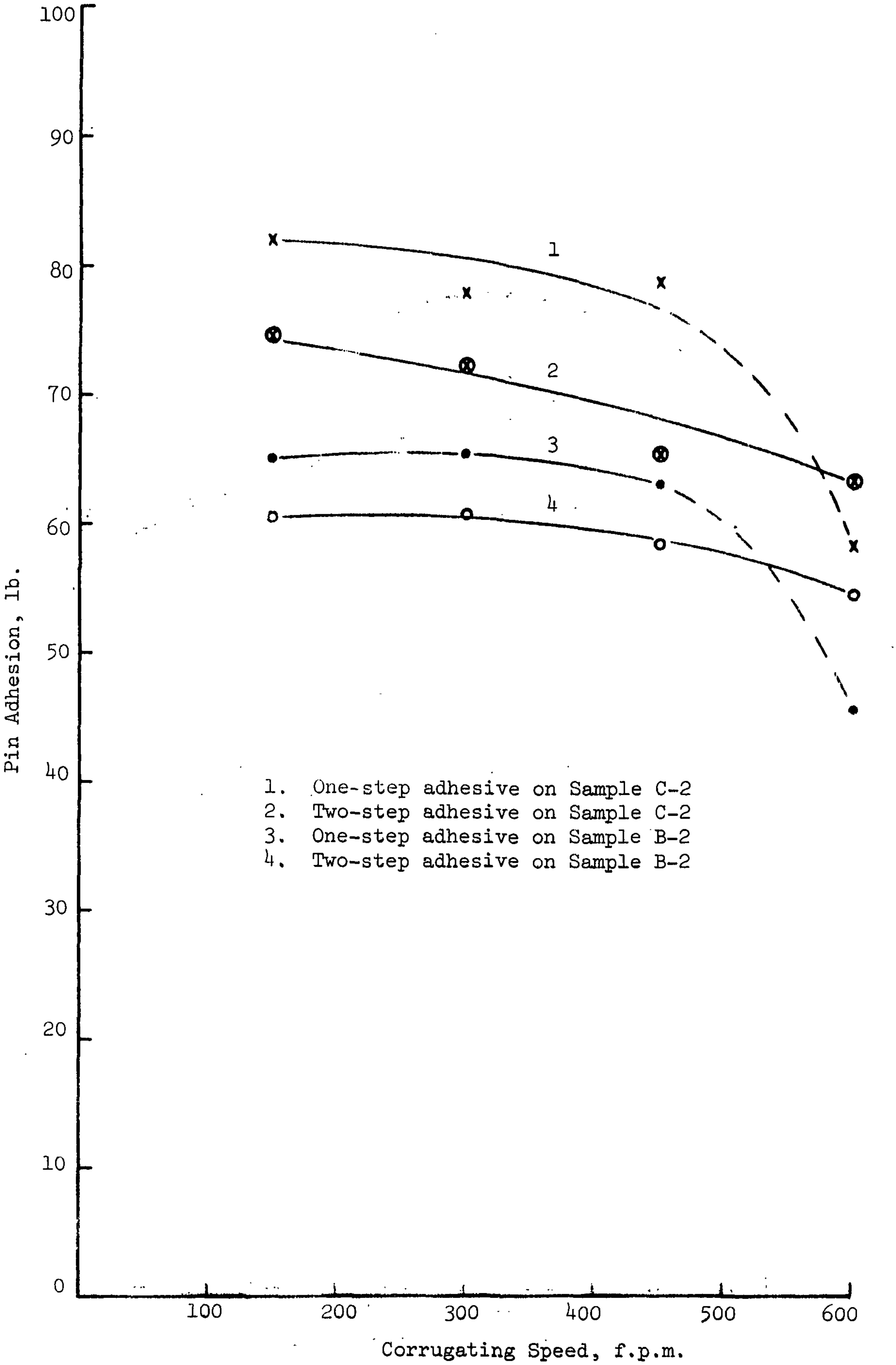

Figure 20. A Comparison of Corrugating Adhesives on Commercial Medium Under Optimum Surface Conditions

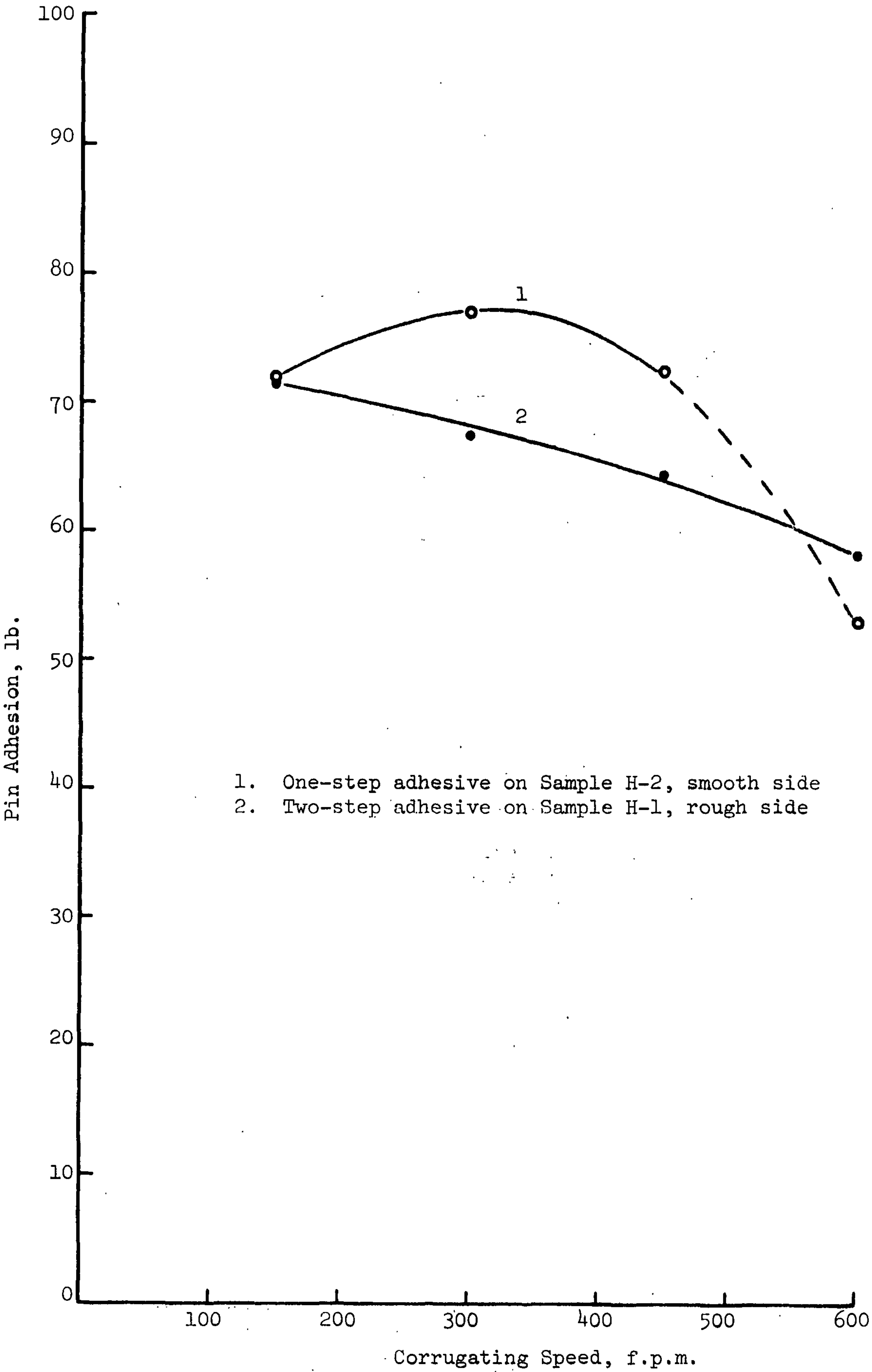

Figure 21. A Comparison of Corrugating Adhesives on Handsheets Under Optimum Surface Conditions

DISCUSSION OF RESULTS

The test data for the experimental medium in Table IV show some differences in physical properties from those listed in Tables II and III but these are due to several factors including slight changes in the supply of commercial medium, changes in the amounts of treating agents, and the tendency for sizing from Aquapel to increase with time. The latter is believed to be the cause for the unexpectedly high water drop values in Samples B-2 and B-3. In general, each unit or series provided a wide range in receptivity. The handsheets proved to be more receptive in most cases considering the contact angle data as well as the water drop values. Mediums "B" and "H" provided relatively smooth and rough surfaces although the differences in the "B" series were not as pronounced as anticipated. It should be pointed out that both sides of Medium "C" were equally rough (3000+ seconds) and, hence, only one side was tested. As would be expected, differences in porosity within a given series were relatively small but a range from 426 to 1840 ml./min. was provided considering the three units. The surface bonding strength results indicate that the treatments with surfactant and sizing agent did not reduce strength which may have influenced the pin adhesion values.

The pin adhesion results presented in Table V and in Fig. 1-21 reflect more or less predicted trends. Optimum pin adhesion was generally attained at low-to-intermediate water drop values or, in other words, on moderately receptive surfaces (Samples B-2, C-2, H-1, and H-2). The differences in adhesive strength between the most receptive and moderately receptive surfaces were frequently quite marginal but at least some advantage for the moderately receptive surface is indicated in eight of ten comparisons (Fig. 1-6 and 9 and 10). The exception was found in the handsheets using the conventional starch adhesive in which case the most receptive surface (H-1) appears to have some advantage. In several cases, medium having water drop values in the range of 400-550 seconds provided pin adhesion values as high as those having much more receptive surfaces (Fig. 2, 3, and 6). However, without exception, the highly sized medium (600+ seconds) produced the lowest pin adhesion values.

The pin adhesion — water drop relationships presented in Fig. 11-16 indicate that the optimum receptivities for the three mediums may differ slightly from those provided by the experimental mediums. The optimum water drop values for Mediums "B" and "H" appear to be reasonably close to those provided by Samples B-2, H-1, and H-2; however, the optimum for Medium "C" appears to be somewhat higher than that provided by Sample C-2. On the basis of the plotted data optimum water drop values of 45, 100, and 250 seconds were selected for Mediums "H," "C," and "B," respectively. The optimum value for Medium "H" is quite arbitrary since pin adhesion does not seem to change appreciably as a function of receptivity suggesting that a wider range in receptivity can be tolerated in a porous medium. The optimum value for Medium H represents an average for Sample H-1, rough side, and H-2, smooth side, since these were optimum for the two adhesives. If these water drop values are plotted as a function of porosity as in Fig. 17 it becomes apparent that the optimum receptivity increases with porosity. It is recognized, of course, that this relationship would probably not hold at extremely high porosities and water drop values. The relationship is at best approximate but at least there is some evidence to suggest that the optimum water drop value for a highly porous medium may be of the order of several hundred seconds, whereas a medium of low porosity such as that provided in the handsheets requires a more receptive surface to reach optimum adhesion. This would seem reasonable considering the idealized rate of penetration equation

$$\frac{d\ell}{dt} = \frac{\gamma r \cos\theta}{4\eta\ell} \qquad (1)$$

where $d\ell/dt$ is the time rate of penetration of a liquid having a surface tension, γ, and viscosity, η, into a porous surface having pore size, r, and where θ is the contact angle formed by the liquid in question and ℓ is the depth of penetration. From Equation (1) it can be seen that the rate of penetration is proportional to the product of the pore size and contact angle. Hence, as pore size decreases, $\cos\theta$ must increase to maintain the same penetration rate when γ, η, and ℓ are constants. Since $\cos\theta$ increases with decrease in contact angle it would be expected that lower contact angles and water drop values would be required on low porosity medium than on medium of high porosity.

In cases where relatively smooth and rough surfaces were available as with Mediums "B" and "H" the smooth surfaces generally provided the better adhesion. This includes comparisons made in the preliminary studies as shown in Tables I and II and it is again indicated in Fig. 18 and 19 where smooth and rough surfaces are compared under conditions of optimum or near optimum receptivities. In this case, the smooth surface provided the better adhesion in three of four comparisons. The exception was again obtained with the handsheets using the conventional starch adhesive where marginally better bonding was obtained on the rough side at corrugating speeds up to 450 f.p.m. Reasons for the advantage shown by the rougher surface in this case are not apparent but it should be borne in mind that the differences in some cases were quite marginal and the terms "rough" and "smooth" are used on a relative basis in this study. The "smooth" sides of the corrugating mediums used in the program are considerably rougher than those of most papers and it has not been established that an extremely smooth surface would be desirable. Mention should be made that smoothness attained by calendering a rough medium was previously found (2) to produce notable reductions in surface bonding strength and could thereby lead to reduced pin adhesion values.

Comparison of the two adhesives shows a general advantage for the one-step adhesive at corrugating speeds up to 450 f.p.m. In fact, in some cases, highly sized medium bonded with the one-step adhesive provided pin adhesion values equivalent to those produced by the conventional two-step adhesive under more favorable receptivity conditions. The advantage provided by the one-step adhesive is indicated in Fig. 20 and 21 where comparisons are made under optimum surface conditions with respect to receptivity and smoothness. The one-step adhesive did not maintain an advantage at the highest corrugating speed but this is believed to be due to the comparatively high gel point of the adhesive used in the present series. Apparently, at 600 f.p.m., the time of contact was not sufficient to allow the temperature to reach the gel point and the resulting bond strength was low. The difference in contact time at 450 and 600 f.p.m. on the Institute's corrugator is a matter of only 0.01 to 0.02 sec. and presumably a slight adjustment in the alkali concentration could reduce the gel point sufficiently to compensate for the difference.

In review, the results obtained in the present study indicate that a relatively smooth, moderately receptive medium should provide optimum pin adhesion under average porosity conditions. The data suggest that slightly more receptive surfaces would be required at low porosities whereas somewhat lower receptivities can be tolerated and may, in fact, be desirable at high porosities. A one-step starch adhesive was found to provide a consistent advantage over the conventional two-step adhesive at corrugating speeds up to 450 f.p.m. and it is assumed that an advantage could be maintained at higher speeds by adjusting the gel point.

These results are in general agreement with those predicted on the basis of the more fundamental study of adhesion properties (1, 2) which suggested that neither a highly sized nor a completely wettable medium would provide optimum adhesion due either to poor acceptance of the adhesive or excessive loss of water. Smoothness was also indicated to be a desirable property because the surface irregularities in a smooth surface were filled with adhesive to a greater extent than those in the rough surface. Finally, a starch adhesive containing moderately and more uniformly gelled particles was suggested since such particles should be better retained on rough surfaces and should not lose water as readily as the conventional starch adhesive. In effect, an adhesive of this type should provide an advantage over a wider range in receptivities. The one-step starch adhesive used in the current program approached these properties and conditions. Further work with starch adhesives would appear to be warranted.

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